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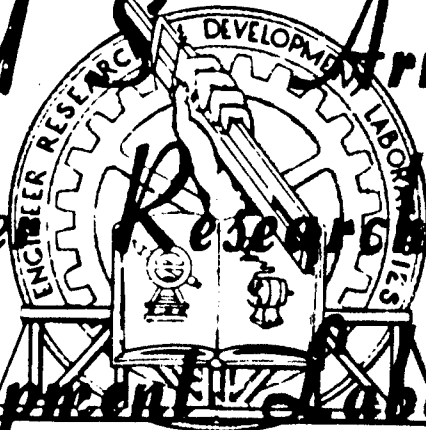
WATER DEMINERALIZATION AND DECONTAMINATION
STUDIES UTILIZING ELECTRODIALYSIS

Task 8M75-05-001-06

9 May 1961

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The Director
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PREFACE

The investigation covered by this report was conducted by the Sanitary Sciences Branch, United States Army Engineer Research and Development Laboratories (USAERDL), under authority of Corps of Engineers Task 8M75-05-001-06 (formerly designated 8-75-05-013). A copy of the project card appears in Appendix A. The preliminary studies were made at the Sanitary Sciences Branch, USAERDL, Fort Belvoir, Virginia; while the actual field tests were conducted at Jackass Flats, the Atomic Energy Commission's Nevada Test Site, Mercury, Nevada.

The period covered by this report was August 1958 through November 1959.

The following personnel were responsible for the supervision of the test program:

Project Engineer	Paul E. DesRosiers, Jr.
Research Section Chief	Don C. Lindsten
Sanitary Sciences Branch Chief	Richard P. Schmitt
Military Department Chief	Neil K. Dickinson

The field test team responsible for installing the equipment and conducting the experiments was as follows:

Project Engineer	Paul E. DesRosiers, Jr.
Equipment Operator	M/Sgt Harvey Fox
Equipment Operator	Frederick W. Wolfes

A glossary of terms as defined for the purposes of this report is included on page 59.

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SUMMARY

This report covers laboratory and field tests of an electro-dialysis process for removal of ionized salts from brackish waters. An experimental unit, manufactured by Ionics, Inc., Cambridge, Massachusetts, was used for the purpose of this study.

Laboratory tests were conducted at Fort Belvoir to determine the following:

- (1) Suitability of operational features to military usage
- (2) Performance characteristics of the process
- (3) Effect of changes in temperature, pH, and feed salinity on performance.
- (4) Factors reducing effectiveness of the ion-selective membranes.

Tests were conducted in Nevada to determine the behavior of the demineralizer in the field when removing dissolved mineral matter from a brackish well water and to determine the capability of the unit for removal of soluble radioactivity from water.

The report concludes:

- a. The electrodialysis process (batch type) is a more economical process than either ion exchange or vapor-compression distillation for removing dissolved minerals from a brackish water containing 3,500 mg/l TDS down to a drinkable level of 500 mg/l TDS.
- b. Dissolved radioisotopes can be removed from water in high percentages by operation of the unit for complete demineralization, but it would not be practicable to do this from a military point of view, since the production rate would decrease sharply and concentration polarization may become significant.
- c. Further laboratory and field tests using the electrodialysis process are necessary in order to develop simplified operating techniques, optimize performance, and adapt the process to military usage.

WATER DEMINERALIZATION AND DECONTAMINATION

STUDIES UTILIZING ELECTRODIALYSIS

I. INTRODUCTION

1. Subject. This report covers laboratory and field tests of an electrodialysis process for removal of ionized salts from brackish waters. An experimental unit, manufactured by Ionics, Inc., Cambridge, Massachusetts, was used for the purpose of these studies.

Laboratory tests were conducted at Fort Belvoir to determine the following:

- a. Suitability of operational features to military usage
- b. Performance characteristics of process.
- c. Effect of changes in temperature, pH, and feed salinity on performance.
- d. Factors reducing effectiveness of the ion-selective membranes.

Tests were conducted in Nevada to determine the behavior of the demineralizer in the field when removing dissolved mineral matter from a brackish well water and to determine the capability of the unit for removal of soluble radioactivity from water.

2. Background and Previous Investigation. In recent years, much interest has been directed toward new processes for production of drinking water from saline water. Intensive studies have been undertaken by the Office of Saline Water, Department of the Interior. The Army uses the vapor compression distillation method for obtaining potable water from the sea. This method is satisfactory on sea water, but it is not economical on most brackish waters because of the inherent scaling problems. Electrodialysis in its present state is not yet economical on sea water. It does, however, present a potentially economical process for demineralization of most brackish waters.

Late in the nineteenth century, biochemists hypothesized the ion-selective or perm-selective membrane, but not until industry had developed ion-exchange resins did the perm-selective membranes become available. The membranes of that time were of the small, fragile type having a high electrical resistance and little chemical resistance.

During recent years, major technical advances have been made in the electric membrane process, and it is becoming more feasible as a method for removal of soluble salts from brackish waters. Research and development has been carried out in the Union of South Africa (1), the Netherlands (2), Great Britain (3, 4), and the United States (5-9) on the electro dialytic removal of salt from saline water sources.

An experimental plant constructed by the British in Tobruk, Libya, has a capacity of 2,400 gallons of water per day reducing a saline water containing an initial concentration of 3,400 mg/l as NaCl to a level of approximately 500 mg/l as NaCl. Membranes 60 by 16 inches were used in this unit which could accommodate up to four hundred 1.3-mm thick membranes in one stack. The membranes were made by the Dutch C.T.I. - T.N.O.¹ A typical experimental run on Tobruk water is summarized in Table I.

Table I. Genuine Tobruk Water-Initial Volume,
73 Gallons (3)

Time Mins.	Volts	Amps	Concentrate Concentration p.p.m. as NaCl by Salinometer	Diluate Concentration p.p.m. as NaCl by Salinometer	Temp.
0	50	13.6	10,300	3400	43°C.
5	50	12.3	10,300	3000	
10	50	11.5	10,500	2650	
15	50	10.5	10,500	2200	
20	50	9.7	10,500	1900	
25	50	8.6	10,500	1600	
30	50	7.5	11,000	1300	
35	50	6.5	10,900	1100	
40	50	5.7	10,800	880	
45	50	5.0	10,600	700	
50	50	4.3	10,600	590	
53.5	50	3.9	10,600	500	

Final Volume Diluate 72 gal: Kwh used (by meter) 0.335.

Throughput 80 gal./hr. Power Consumption 4.65 Kwh./1000 gal.

Analysis of treated water:

NaCl 386 p.p.m.

Total Hardness 58 p.p.m. Power consumption
(graphical) 5.08 Kwh./1000 gal.

1. Central Technical Institute of the Organization for Applied Scientific Research in the Netherlands - Toegepast Natuurwetenschappelijk Onderzoek.

In mid-1953, in the Union of South Africa, work began to develop special membranes that could be suitably used for desalting of certain brackish waters. From January 1954 to July 1955, a 50-cell, 250-gph, electrodialysis unit was in operation at Pretoria. By September 1957, the final designs were completed for the 2.4-mgd unit in the Free State Geduld Mine District. The membranes were 84 3/4 by 25 3/4 inches of the parchment type (1).

In Wichita Falls, Texas, an experimental four-stack electric membrane demineralizer was installed in September 1954. The unit was the first such operation of its size in the United States. It was used as an auxiliary to the power plant and also as an experimental unit in water supply. The electrodialysis unit treated approximately 2,400 gph (10).

In June 1955, tests of an electric-membrane demineralizer were conducted by the Department of the Navy. The unit was investigated for desalting of sea water with particular emphasis for use aboard submarines in lieu of current vaporization processes. Table II summarizes data taken from the Navy's report on the investigation.

Table II. Test Results with Sea Water
Electrodialysis Demineralizer (11)

Characteristic	Results		
Type of feed	Sea water	Potable water	Sea water
Quality of product	Potable water	Battery water	Battery water
Economy (gal/kw-hr)	5.55	52.90	3.45
Rate output (gph)	15	65	8.7

In 1955, the Office of Saline Water, U. S. Department of the Interior, conducted field tests of an electrodialysis process at Buckeye, Arizona, and at Miller, South Dakota.

Results of the Arizona tests showed that the concentration of dissolved salts in the well water was reduced from 3,830 to 229 mg/l. The results also indicated that all initial constituents of the well water were effectively removed with the exception of silica and boron which showed little or no removal. The unit was equipped with special filtering units installed in the feed-water line to prevent foreign materials from entering the system. These filtering units were found to be desirable in previous tests since if suspended matter were allowed to enter the system the membranes would be fouled.

During the Arizona tests, there was considerable CaCO_3 scaling most of which was found on the anion membranes; however, this difficulty was eliminated by the injection of a small amount of acid into the electrode and brine streams. When the CaSO_4 concentration exceeded the theoretical solubility limit in the brine stream, precipitation occurred. This obstacle was overcome by adjustment of the brine stream flow rate accordingly.

The well water in South Dakota contained 1.3 mg/l of iron. This iron caused sliming of the membranes, particularly the anion membranes. It was found, however, that proper pretreatment (oxidation and filtration) of the feed water was necessary. No further difficulties resulted after these steps were taken.

Results of both tests (Arizona and South Dakota) showed no chemical deterioration in membrane composition at any time. It was found, however, that there was a physical weakening in some membranes due to the inefficient inert backing material used (glass-backed cation membranes). The plastic backed anion membranes showed no loss of strength throughout the tests (12).

II. INVESTIGATION

3 Description of Process. One purpose of the perm-selective membrane demineralizer is to remove ionized salts from brackish waters. The production rate is dependent primarily upon two factors: the amount of dissolved solids in the feed water and the amount of dissolved salts desired in the product effluent. To produce demineralized water from saline water by the electric membrane process, an electric current is passed in series through two saline water streams which flow in parallel in adjacent passages between selective ion-permeable membranes. A schematic diagram of the membrane stack is shown in Fig. 1. This membrane stack consists of alternating anion and cation membranes separated by suitably manifolded sections called "spacers." The stack is provided with two external cathodes made of stainless steel, type 316, and two internal anodes composed of platinum-coated tantalum.² As can be seen in Fig. 1, the positive ions (+) (cations) are attracted to the negative electrode (cathode). The cation membrane (C) will pass only positive ions; anions cannot penetrate this membrane. The negative ions (-) (anions) are attracted to the positive electrode (anode). The anion membrane (A) will pass only anions. This results in a series of alternating dilution and concentration compartments. These compartments are manifolded in such a way as to yield only one stream which is being diluted and one stream which is being concentrated.

2. Hastelloy C used as cathode material and only one internal anode, platinum-coated on both sides, used in Fort Belvoir tests.

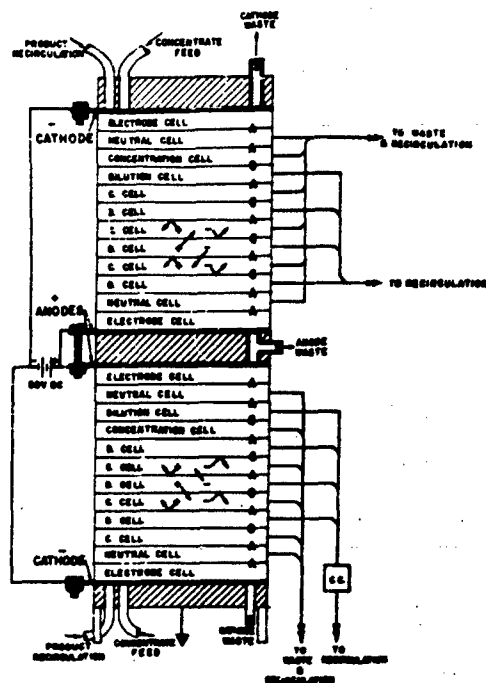


Fig. 1. Cross section of membrane stack, aquamite 30-B electrodialysis demineralization unit.

4. Methods of Analysis and Instrumentation. The following methods of analysis and instrumentation were used:

a. Methods of Analysis.

(1) Chloride. Chloride determinations were performed with 0.10 N AgNO_3 with 5 percent K_2CrO_4 as an indicator at pH 8.3.

(2) Alkalinity. Methyl orange-xylene cyanol was used to determine alkalinity. In alkaline solution, methyl orange-xylene cyanol is green and turns grey just before the end-point is reached. With the addition of a few drops of titrant, 0.02 N H_2SO_4 , the solution turns a pinkish-purple indicative of the end-point.

(3) Total Hardness. An indicator, UniVer I, was used to determine total hardness. The indicator contained

sodium cyanide used to complex interfering iron and copper. A standard solution of ethylenediaminetetraacetate (disodium salt) (EDTA) was used as the titrant. The color change was from a light red to a light blue at the end-point.

(4) Calcium Hardness. CalVer II as an indicator at a pH of approximately 11.3 was used to determine calcium hardness. One milliliter of 8 N KOH was used to approximate this pH. The color change at the end-point was from a light red to a blue.

(5) Sulfate. Barium chloranilate, $\text{BaC}_6\text{Cl}_2\text{O}_4$, was used to determine sulfate. The reaction between solid barium chloranilate and sulfate ion at pH 4 in a 50-percent ethanol-water solution liberates the purple-colored acid-chloranilate ion. The adsorption was carried out at a wave length of 525 m μ , using a Fisher Electrophotometer. Certain cations interfered with the procedure and subsequently were removed by cation-exchange resins in the hydrogen form. This method, found highly successful, was used through the Nevada tests.³

(6) Sodium. Sodium was determined by difference.

(7) Iron, Silica, Aluminum, and Copper. A Hach Chemical Company DR Colorimeter was used to determine iron, silica, aluminum, and copper.

(8) pH. A Bechman pH meter, Model H-2 was used to determine pH.

b. Instrumentation.

(1) Pressure Gauges. Four pressure gauges were used. Two measure the pressure drop across the filters in the feed line. A pressure gauge is installed in each of the two recirculating streams (one in the brine-stream influent and the other in the product-stream influent). The product stream pressure is normally set from 0.5 to 1.0 psig higher than the brine stream pressure.

(2) Flow Meters.

(a) A rotameter-type flow meter measured the input rate of blowdown to the brine recirculation system.

3. More information may be found in Analytical Chemistry, Vol 29, No. 2, Feb 1957, pp. 281-283.

(b) Another rotameter was installed in the product recirculation stream to measure the recirculation rate.

(c) A Fischer-Porter flow meter was used to measure the electrode-stream flow rate. The flow rate was read in centimeters. This reading was referred to a calibration chart to determine the actual flow rate.

(3) Filter. A cellulose-type filter was used in the product recirculation stream in order to remove the oxides of iron, if any, from this stream. This filter proved very useful for removal of iron slime which had been flushed from the stack during operations at Fort Belvoir and Nevada.

(4) Thermometer. A bi-metallic thermometer set in the hydraulic system measured the temperature of the feed water. Temperature had a marked effect upon the performance of the demineralizer. The equation describing this effect is:

$$\text{Product flow rate at } T = \text{Product flow rate at } 70^{\circ} \text{ F.} \\ (T \rightarrow 120^{\circ} \text{ F}) \quad \times [1 + 0.011 (T - 70)]$$

(5) Conductivity Cells.

(a) The conductivity cell controls the product recirculation in such a way that the "batch" is discharged only at the product concentration previously set on the conductivity dial located on the instrument panel. The setting on this dial does not necessarily indicate the correct product concentration and, therefore, must be calibrated prior to operation of the demineralizer.

(b) Another conductivity meter, Barnstead Purity Meter, was used to determine the total dissolved solids content of the effluent product water during the decontamination studies conducted in Nevada. The scale of the meter is calibrated from 0 to 15 ppm expressed as sodium chloride and has a temperature-correction scale with a range of 64° to 190° F.

(6) Watthour Meters.

(a) An a-c watthour meter with cyclometer register was used to record the total power consumption of the unit during the Nevada field tests.

(b) A d-c, switchboard-type, watthour meter was used to record power consumed by the membrane stack during the field tests.

(7) Ammeters. Two d-c ammeters were used to measure the current in the top and bottom of the membrane stack, and one d-c voltmeter was used to measure the voltage impressed upon the stack. The range of the ammeters was 0 to 10 amps and could be read to plus or minus 0.1 amp. Another d-c ammeter, therefore, was used for the low ranges since most currents in the Nevada field study were in the 0- to 0.7-amp range. This ammeter had a range of 0 to 1.000 amp and could be read to plus or minus 0.005 amp.

(8) Scaler. A Nuclear-Chicago Scaler and Geiger-Mueller end window tube assembly was used to count cesium beta activity during decontamination studies at the Nevada Test Site.

(9) Acid Pump. A corrosion-resistant duplex proportioning pump with built-in control dials was used to feed acid to both the brine and electrode streams.

5. Laboratory Studies. The following is a description of laboratory studies made at the Sanitary Sciences Branch, Fort Belvoir.

a. Description of Equipment. Figure 2 is a simplified schematic flow sheet of the unit. The feed water splits into two

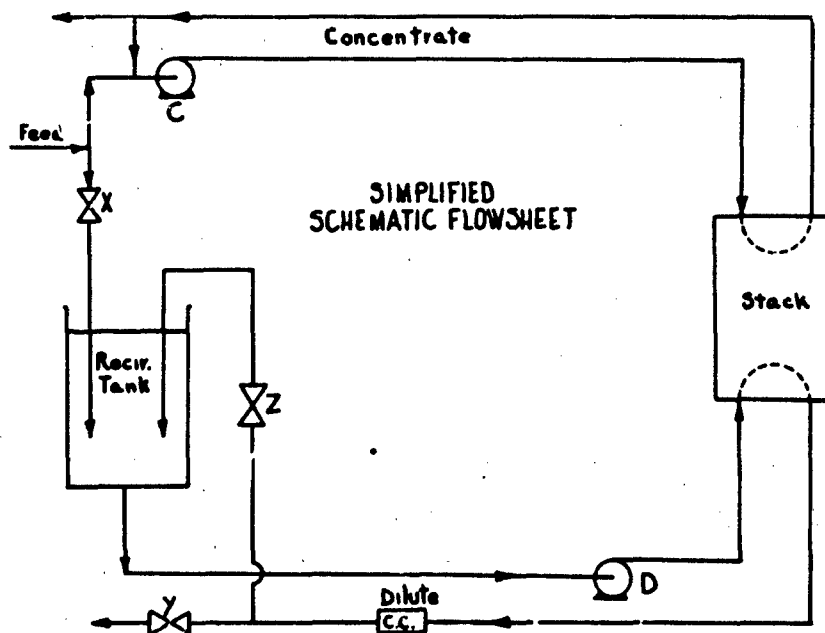


Fig. 2. Simplified schematic flow sheet of unit.

streams; one is continuously fed into the brine stream, and the other is fed intermittently to the recirculation tank to restart a new cycle. The concentrate pump (C) recirculates the brine stream through the stack. A volume of brine equal to that of the feed water added is pumped out of this system as waste or brine effluent.

After the feed stream has filled the recirculation tank, valve X shuts automatically prohibiting further flow to the tank. This feed water batch is recirculated through the stack by means of pump D, through the conductivity cell CC, and through solenoid-valve Z to the tank. When the conductivity cell CC indicates the desired salinity of the final effluent, valve Z automatically closes and valve Y opens allowing the product to discharge to a suitable storage system. When the recirculation tank empties, valve X opens and raw water again passes through the system. When raw water reaches the conductivity cell CC, valve Y closes and valve Z opens. Valve X closes when the recirculation tank is full. Raw water then recirculates until a satisfactory product is obtained. Hence, there is an intermittent discharge of each individual batch of water since the degree of recirculation is determined by the total dissolved solids content of the raw water and concentration of the product.

Figure 3 is a diagrammatic representation of the major flow system involved in the demineralization cycle. This flow diagram, unlike Fig. 2, contains both electrode flow and acid injection systems. The electrode stream is constantly acidified with a solution of NaHSO_4 which becomes an acid by ionization. A high concentration of hydroxide ions is formed at the cathode (equation a(2), paragraph 7, p. 47) and must, therefore, be neutralized by acid or a scale problem will develop in most cases. The concentration and rate of feed of acid is dependent upon two factors: (1) the concentration of the acid-consuming ions in the feed water and (2) the applied d-c voltage. The electrode stream (cathode plus anode wastes) contains hydrogen, oxygen, and chlorine gases which must be suitably vented and discharged.

Figure 3 shows that the acid is also injected into the brine (concentrate) stream. This acid is necessary in order to reduce the alkalinity present in this stream, since a reduction of the salt concentration in the product stream is also accompanied by a proportional increase in the waste stream. The concentration and rate of acid feed is dependent upon the total dissolved solids, pH, calcium, and bicarbonate concentration of the brine blowdown stream and its temperature. From these data, a Langelier Index (see paragraph 7) is calculated and acid is fed accordingly.

If the concentration of the feed water contains appreciable amounts of calcium and sulfate ions, scaling conditions may

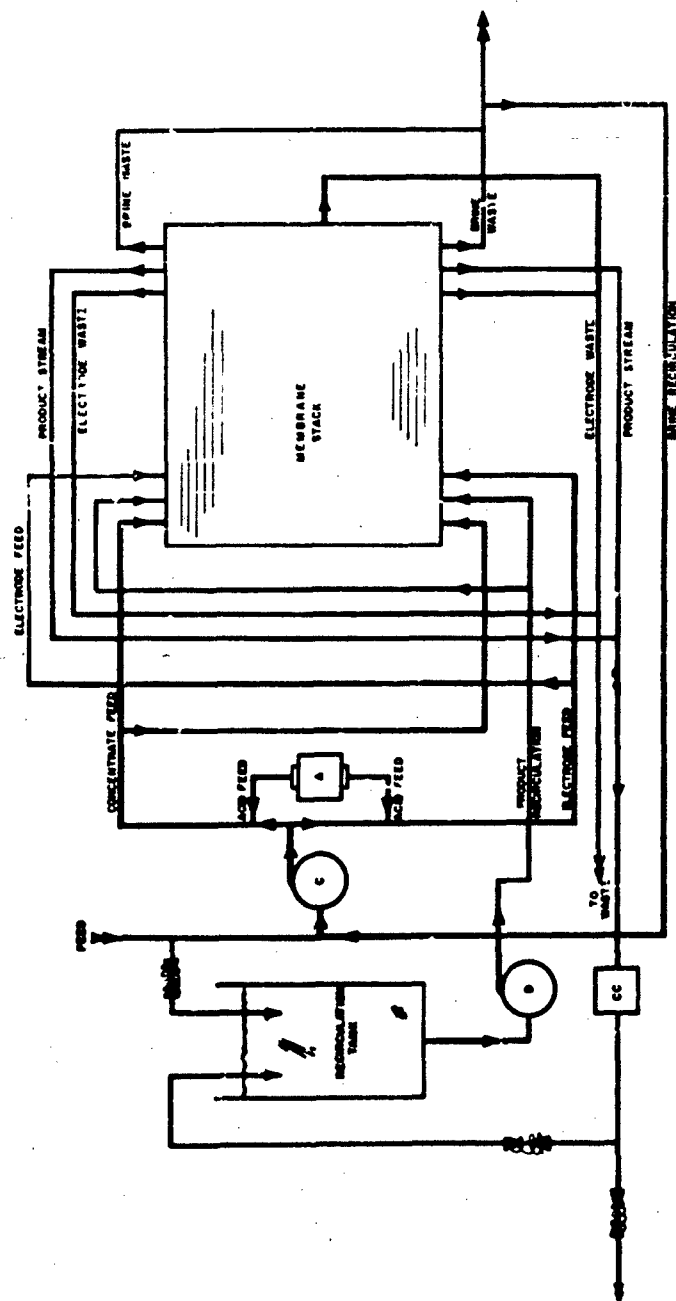


Fig. 3. Flow diagram, electrodialysis demineralization unit.

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exist and calcium sulfate may precipitate and eventually cause the blockage of the flow paths in the cells. This calcium sulfate scale formation can be avoided by proper adjustment of the brine-stream blowdown rate. This will prevent the brine waste stream from becoming saturated with calcium sulfate and will avoid precipitation.

Figure 4 shows the electrodialysis unit assembled for test in the laboratory at Fort Belvoir.



E12130

Fig. 4. Model 30-B electrodialysis unit at Fort Belvoir.

One-hundred-fifteen-volt, 60-cycle, single-phase power was supplied to the system. A rectifier produced the d-c voltage required by the demineralization process. The brine, feed, acid, and product pumps were all wired for 115-volt, 60-cycle, single-phase operation. All instruments required the same a-c voltage. The entire electrodialysis unit was thoroughly grounded. The membrane stack consisted of 100 cell-pairs (each cell-pair contains one cation, one anion, and two spacers) (Fig. 5). The membranes were 9 by 10 inches and were 0.6 mm thick. The cation membranes were constructed of sulfonated divinylbenzene polystyrene impregnated on fiberglass backing, and the anion membranes were made of quaternized divinylbenzene pyridene impregnated on dynel cloth. The membrane spacers were specially constructed flow channels of polyvinylchloride. Figures 6 and 7 are magnified photos of both cation and anion membranes.



F11112

Fig. 5. From left to right: anion membrane, membrane spacer, and cation membrane.

b. Tests and Test Results. A series of tests was conducted on the electrodialysis unit from August to October 1958. The total operating time was 204 hours. The purpose of early tests was to develop a thorough familiarization of operating procedures. A 450-gallon wooden tank, approximately 58 inches high and 49.5 inches inside diameter, was used as the container for the synthetic make-up water source.

Three tests were performed on different synthetic waters.

(1) The concentration of the initial make-up water was 3,134 mg/l NaCl. Analysis was as follows:

Turbidity	- 22 units
Total alkalinity	- 29.3 mg/l as CaCO_3
Total hardness	- 83 " " "
Ca hardness	- 67 " " "
Mg hardness	- 16 " " "
Salinity	- 3134 mg/l as NaCl
Aluminum	- 0.43 mg/l
Copper	- 0.28 "
pH	- 5.4

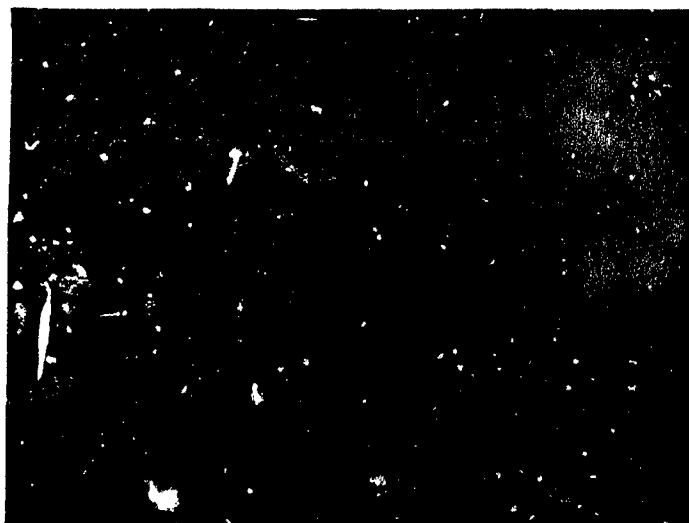


Fig. 6. Cation membrane (magnified 13 times).

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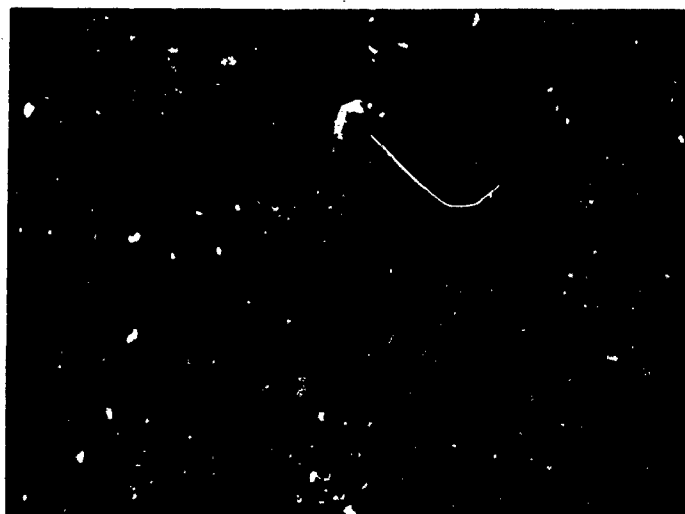


Fig. 7. Anion membrane (magnified 13 times).

G1493

This water was fed to the unit at a rate of 0.4 gpm. The average production rate was 40.4 gph at a feed-water temperature of 75° F. The unit was operated for 6 hours. Operating data for this run were as follows:

Conductivity setting	- 675 μ mhos/cm
Make-up feed to brine stream-	0.4 gpm
pH cathode effluent	- 3.1
Acid feed, 12% NaHSO ₄	- 0.28 gph
Salinity of product	- 463 mg/l
Voltage at start of cycle	- 50.5 volts
Voltage at end of cycle	- 52.5 volts
Current at start of cycle	- 2.4 amps
Current at end of cycle	- 1.18 amps

(2) On 7 August, the second test began. Analysis of feed water was as follows:

Salinity	- 4873 mg/l as NaCl
Turbidity	- 10 units
Total alkalinity	- 72 mg/l as CaCO ₃
Total hardness	- 66.0 " " "
Mg hardness	- 9.4 " " "
Aluminum	- 0.30 mg/l
Copper	- 0.23 "
pH	- 7.5

In this test, a make-up water containing 4,873 mg/l NaCl was fed to the unit at a temperature of 75° F. The following operating data were accumulated from the test:

Conductivity setting	- 675 μ mhos/cm
Make up feed to brine stream-	0.4 gpm
Acid feed, 12% NaHSO ₄	- 0.28 gph
Voltage at start of cycle	- 50 volts
Voltage at end of cycle	- 52 volts
Current at start of cycle	- 2.8 amps
Current at end of cycle	- 1.2 amps
Product rate	- 27 gph
Salinity of product	- 469 mg/l as NaCl
Turbidity of product	- 0
pH of product	- 7.4
Operating time	- 6 hours

.3) The feed water analysis for the third test was as follows:

Salinity	- 5067 mg/l as NaCl
Total alkalinity	- 32.8 mg/l as CaCO ₃

Phenol alkalinity	- 3.2 mg/l as CaCO_3
Total hardness	- 74.6 " " "
Ca hardness	- 62.6 " " "
Mg hardness	- 12.0 " " "
Aluminum	- 0.16 mg/l
Copper	- 0.15 "
pH	- 8.4

The total operating time for the third test (conducted from 25 August to 27 August 1956) was approximately 26 hours. The purpose of this test was to calibrate the conductivity control (Fig. 8 lower left, on panel board) in order to determine what setting would correspond to a product salinity of 500 mg/l. It was found that a setting of 800 μ mhos/cm on the conductivity meter would give the desired product concentration. The average production rate during this test was 26.7 gph at 78° F. The product water contained 505 mg/l as NaCl, with a pH of 7.2.



F11118

Fig. 8. Control panel of electrodialysis unit.

On 20 August, prior to the third test, a 2,500-gallon, rubber-lined tank was used in conjunction with the 450-gallon feed tank. This added capacity insured a longer operating time. A sufficient quantity of sodium chloride (granular) was dissolved into this tank to give a concentration of about 5,000 mg/l as NaCl. A new hydraulic system was installed which enabled the make-up water to be recirculated from the 2,500-gallon tank to the 450-gallon tank and vice versa until a uniform, homogeneous, feed-

water concentration was established. The pH of the feed water was raised from 5.4 to 8.3 with sodium hydroxide.

On 3 September, a new synthetic water containing the following analysis was made up:

Salinity	- 9,004 mg/l as NaCl
Total alkalinity	- 32 Mg/l as CaCO_3
Phenol. alkalinity	- 2 " " "
Total hardness	- 99 " " "
Ca hardness	- 87 " " "
Mg hardness	- 12 " " "
pH	- 8.6

The unit was operated about 32 hours on the above water, from 4 to 9 September 1958, without any appreciable difficulties other than a slow drop in production rate. The following data represent a typical run on the 9,004 mg/l water:

Conductivity setting	- 800 μ mhos/cm
pH cathode stream	- 2.4
Acid feed, 2% NaHSO_4	- 0.13 gph
Make-up feed to brine stream	- 0.4 gpm
Brine blowdown rate	- 20.5 gph
Brine blowdown salinity	- 13,668 mg/l as NaCl
Voltage at start of cycle	- 50 volts
Voltage at end of cycle	- 52.3 volts
Current at start of cycle	- 3.45 amps
Current at end of cycle	- 1.33 amps
Product salinity (79° F)	- 490 mg/l as NaCl
pH of product	- 7.1
Product rate	- 15.4 gph

On 9 September, the production rate fell to 14.2 gph. The pressure drop across the inlet filters (Fig. 9, right-center) was 15 psi. The unit was shut down, and the filter cartridges were inspected. Filter elements were caked with a brown, mud-like substance; this substance was analyzed qualitatively and found to contain a considerable amount of iron. The two filter cartridges (5 micron pore diameter) were replaced by new ones.

On 11 September, 1 pound of citric acid was added to the 450-gallon tank which contained 2,830 mg/l NaCl. The pH of this water was 3.3. The unit was operated with the rectifier off. The contents of the tank was fed to the electrodialysis unit to flush the unit of any iron deposits in the hydraulic system and membrane cells. This acid-flushing improved the conductance of the membranes while removing most of the iron deposits.



F11115

Fig. 9. Side view of unit showing filtering system.

A new feed-water solution was made up and was fed to the unit on 16 September. Following is an analysis of that feed water:

Salinity	- 7078 mg/l as NaCl
Total alkalinity	- 38.6 mg/l as CaCO_3
Phenol. alkalinity	- 2.0 " " "
Total hardness	- 84 " " "
Ca hardness	- 72 " " "
Mg hardness	- 12 " " "
pH	- 8.4

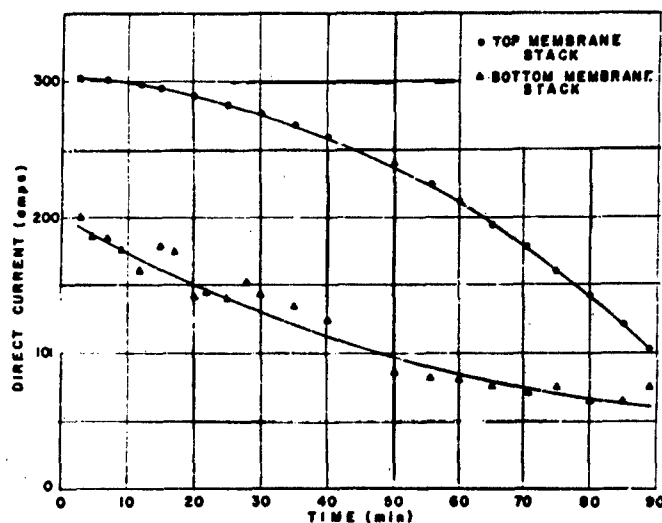
The unit operated satisfactorily from 16 to 19 September, except for leakage from the bottom of the cathode area. Plastic gasket compound was put around the cathode area but did not provide the watertightness desired. The tie-rod nuts were then screwed down tighter but the leakage continued and became progressively worse. The main trouble was in the Hastelloy C cathode (Fig. 10, right) where the tie-rod holes were punched and metal "stretched" which gave an "oil

caning" effect to the metal. When the tie-rod nuts were tightened, the cathode "warped" and a space was left between the cathode and the electrode gasket causing the major leaks. This problem was later (prior to Nevada test) remedied through the use of 1/32-inch, stainless-steel, type 316 sheet for the cathode material. This electrode material did not stretch when the holes were punched. On 19 September, the production rate remained constant at 19.0 gph until the fifth cycle, when it dropped to 18.9 gph; during the sixth cycle, however, abnormal stack current readings were noted. The production rate fell to a low of 13.7 gph.



Fig. 10. Electrodes used in unit: left, anode; right, cathode. F11113

As shown in Fig. 11, at the start of the cycle, the current in the top of the stack was 3.02 amps, while that in the bottom of the stack was 2.00 amps. This fact indicated that something was wrong since under normal conditions, the currents are equal. The graph also indicates that the current in the top of the stack decreased uniformly with respect to time, but the current in the bottom of the stack was erratic as it decreased. This indicated that there was unusual electrical resistance in the bottom of the stack causing a decrease in amperage. The membrane stack was disassembled, and each membrane and spacer was physically inspected. The membranes in the top half of the stack were in fair condition. Some were dry and warped near the outside edges, while the spacers were white, opaque in color (original color was clear, transparent



F11151

Fig. 11. Effects of spacer degradation causing high resistance in bottom of stack.

vinyl). In the bottom half of the stack, practically all of the spacers were a deep yellow in color, specifically around the "holes" not used for water flow (Fig. 12). These holes in the spacers contained, for the most part, a yellow gelatinous substance resembling sulfur (Note E of Fig. 13). The holes, particularly those used for the feed-water influent, also contained other foreign substances (Fig. 13).

Many of these spacers had literally "melted" (chemical structure and physical appearance of the spacers were changed) in several of the sections, and nine spacers had to be replaced; only 20 replacements were available. The membranes in the bottom of the stack revealed damage and had to be replaced because of impregnation from the previously mentioned foreign substances. The sections under the holes of the spacers were also dried out and wrinkled. The cation membranes were especially damaged due to this melting effect. The reason for the yellowing of the spacers and their subsequent effect on the membranes, particularly the outermost sections near the edges, was a degradation reaction taking place at the holes. There was no liquid flow through these holes, and they had no apparent, significant use. Since the spacers were constructed of a copolymer of vinyl chloride and vinylidene chloride or a variation thereof, chlorine molecules (or chloride ions)

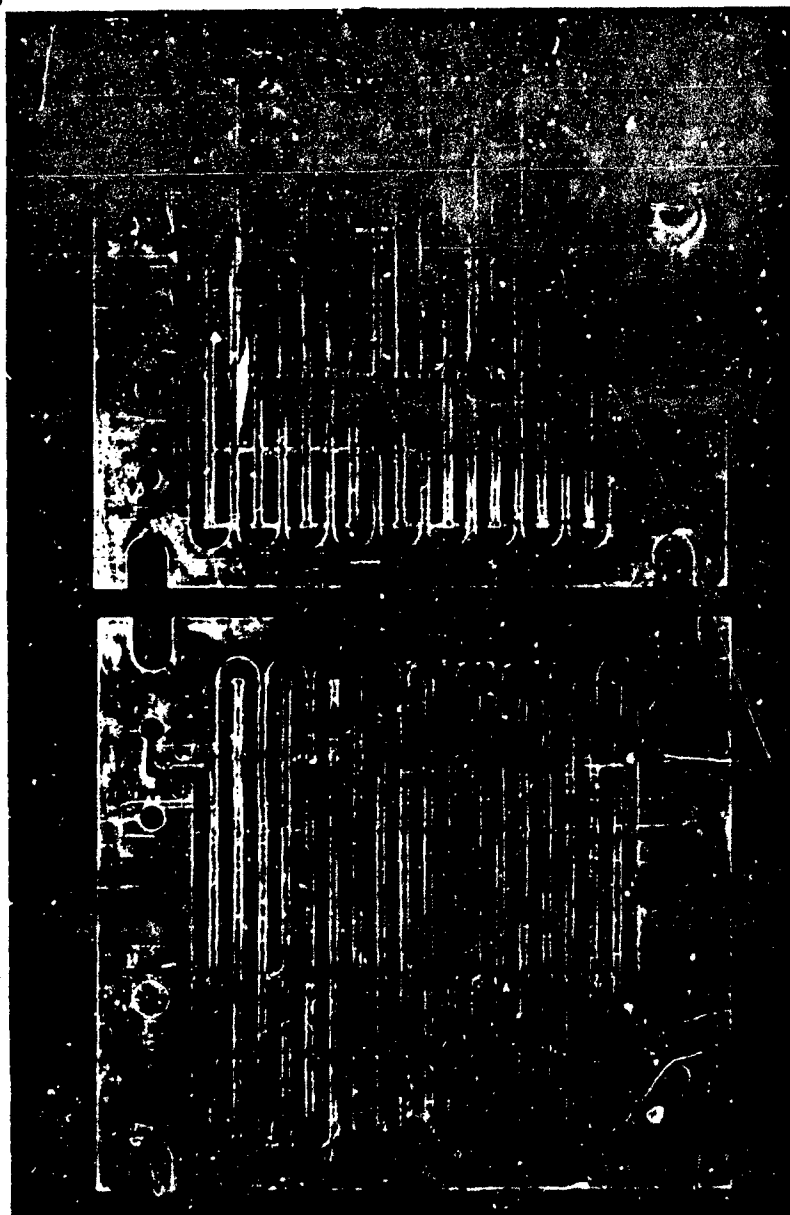
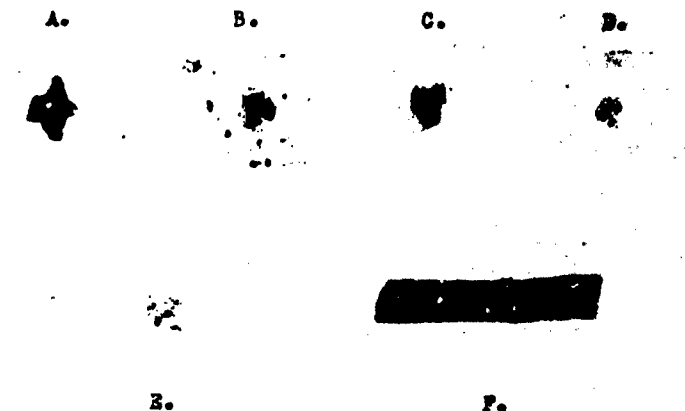


Fig. 12. PVC membrane spacers: top, old type of spacer (four sets of holes); bottom, new type of spacer (two sets of holes); arrow denotes yellowed area. G1330



Rubber

Rust

Metal

Plastic

Degradation Product of Membrane Spacer

Section of Electrode Spacer

01198

Fig. 13. Foreign substances found in membrane stack.

evolved by the electrodialysis process (due to the high current density about these isolated holes) could collect and be retained in the holes. This subsequently initiated the degradation reaction.

Further inspection of the membranes revealed that the anion membrane, particularly those near the neutral cell (Fig. 1), were coated with a thin, brown slime. This slime was found on the dilution side of the anion membranes. The slime, similar to that observed in test work done on 9 September 1958, was analyzed qualitatively and found to contain iron. It was believed that most of the iron entered the system due to failure of the filtering elements in the feed line. It was observed during this test that the

polyethylene feed and discharge lines to and from the stack were filled with turbid water. The membranes were soaked in 0.5 N HCl and scrubbed physically to remove the iron slime.

On 22 September, another cartridge-type filter was added to the unit. This filter was installed in the product recirculation line just ahead of the recirculation tank. This filter clarified the recirculated dilute stream by removal of oxides of iron and other precipitates which resulted as oxidation products in the membrane stack. On 25 September, a rotameter was installed in the same product recirculation line ahead of the new filter to determine the recirculation flow rate. Readings showed that the rate remained at approximately 3.7 gpm.

With new and cleaned membranes and after the above changes were made, the unit was operated on 7,038 mg/l NaCl make-up water. The cycle took 62.5 minutes to give a product salinity of 538 mg/l. The product rate was 18.7 gph. These results are shown in Fig. 14.

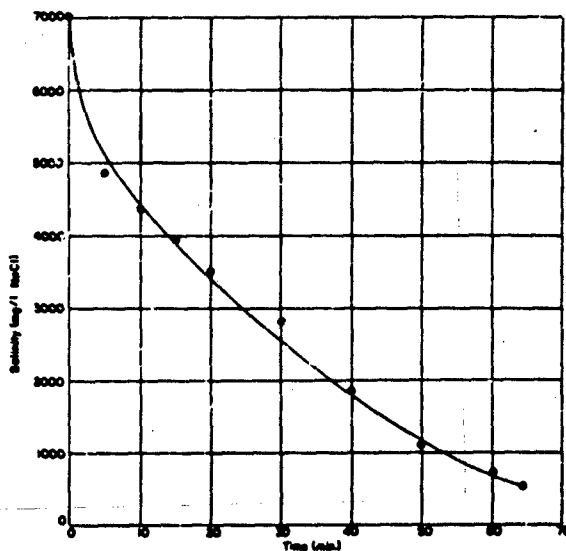


Fig. 14. Typical demineralization cycle.

On 1 October, synthetic water containing 12,070 mg/l NaCl was prepared. Table III shows the results of the tests on this water.

Table III. Synthetic Water Test Results

Characteristic	Influent (Feed)	Effluent (Product)
pH	8.35	7.4
Alkalinity (mg/l CaCO_3):		
Total	44.0	14.8
Phenol.	2.0	0
Hardness (mg/l CaCO_3):		
Total	123.4	2.2
Calcium	112.4	2.2
Magnesium	11.0	0
Salinity (mg/l NaCl)	12,070	497

The final tests with synthetically prepared feed water consisted of three runs on 1,650, 1,770, and 2,515 mg/l NaCl water, respectively. An a-c kilowatt-hour meter was installed in order to measure the total power consumed. The average total power consumption over the range 1,650 to 3,580 mg/l NaCl was 8.5 kw-hr/24-hr day. Maximum probable error was calculated so as to develop a reasonable curve representing the mathematical relationship between power consumption and dissolved solids removed. Figure 15 depicts the variation to be essentially constant through the range.

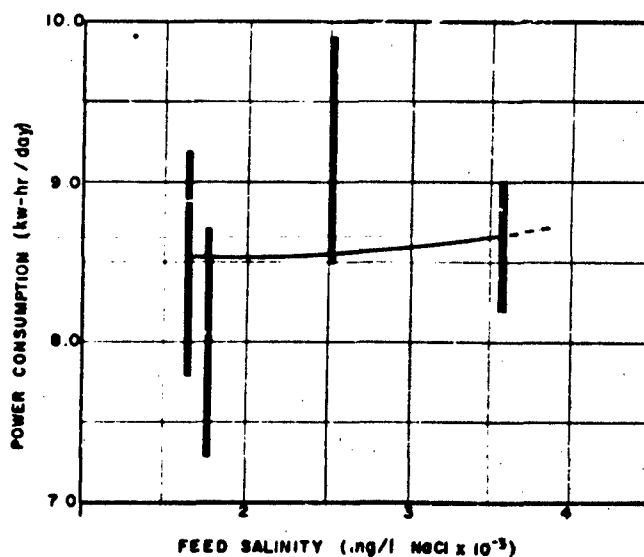


Fig. 15. Effect of salinity on total power consumption.

On 13 October, a test was conducted designed to check variation of electrode (cathode plus anode wastes) stream pH with respect to time. The hydroxide ion concentration of the electrode stream is directly proportional to the amperage through the membrane stack. A rise in the hydroxide ion concentration will result in a rise in the pH of the electrode stream. Hence, an acid (e.g. NaHSO_4) is injected at a constant rate into the electrode stream, the potential hydroxide concentration is reduced; this relationship is shown in Fig. 16.

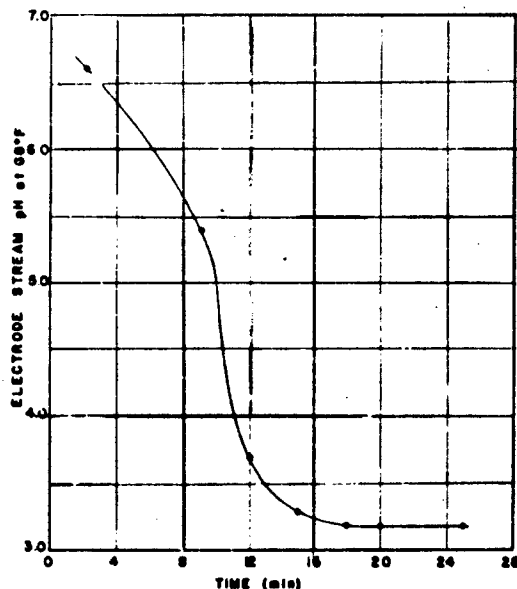


Fig. 16. Behavior of electrode stream pH during a typical cycle.

F11150

Ionics, Inc., gave the following mathematical relationship which explains the effect of temperature on the production rate of the unit:

$$\text{Production rate at } T = \text{Production rate at } 70^{\circ} \text{ F} \times [1 + 0.011 (T - 70)]$$

For each degree rise in feed-water temperature, the production rate will rise 1.1 percent. In the laboratory tests conducted, this relationship was found to hold reasonably true within experimental error.

The change in feed water salinity had a very measurable impact on the production rate, and data obtained in the course of the various laboratory tests is shown in Fig. 17. The plot of feed salinity versus production rate at 70° F shows that the slope changes rapidly from 1,650 to 3,500 mg/l NaCl and is essentially constant from 5,000 to 12,000 mg/l NaCl.

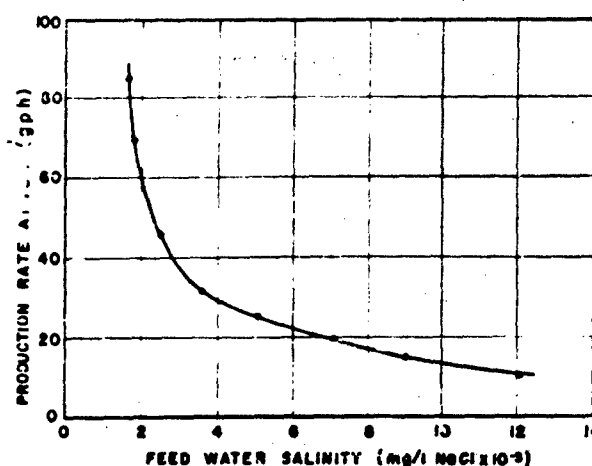


Fig. 17. Effects of feed water salinity on production rate (product water contained 500 mg/l TDS).

6. Field Studies. The following field studies were conducted at Jackass Flats, Nevada Test Site (Figs. 18 and 19). The water used for the studies was obtained from well J-11. The depth of the well was 1,330 feet. The electrodialysis unit was housed in a temporary building (Fig. 18). Well water was pumped to a 500-gallon, GRS-coated nylon tank used as the feed tank for the electrodialysis unit.

a. Description of Equipment. The electrodialysis unit was assembled in a manner similar to the arrangement used for the Fort Belvoir tests, with the exception of an added ammeter which had a range of 0 to 1.000 ampere and could be read to plus or minus 0.005 ampere. This low-range ammeter was necessary since the feed water was demineralized to low levels (1.5 mg/l TDS) during the decontamination tests. The control panel for the unit is shown in Fig. 20. Figure 21 shows the right portion of the unit with the membrane stack at the right.

The membrane stack contained the same type of membranes as those used in the Fort Belvoir tests, the only difference



Fig. 18. Location of electro dialysis unit at test site. F9336



Fig. 19. View of test area, Jackass Flats, Nevada Test Site. Q9270

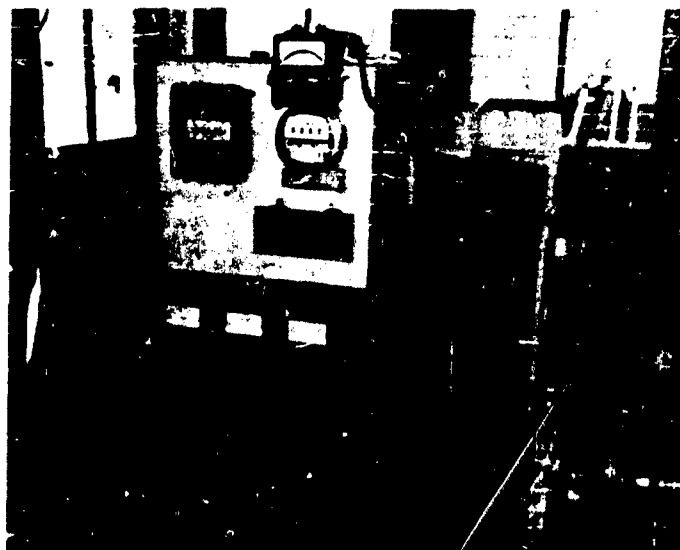


Fig. 20. Control panel with a-c and d-c watt-hour meters and milliammeter.

F9352

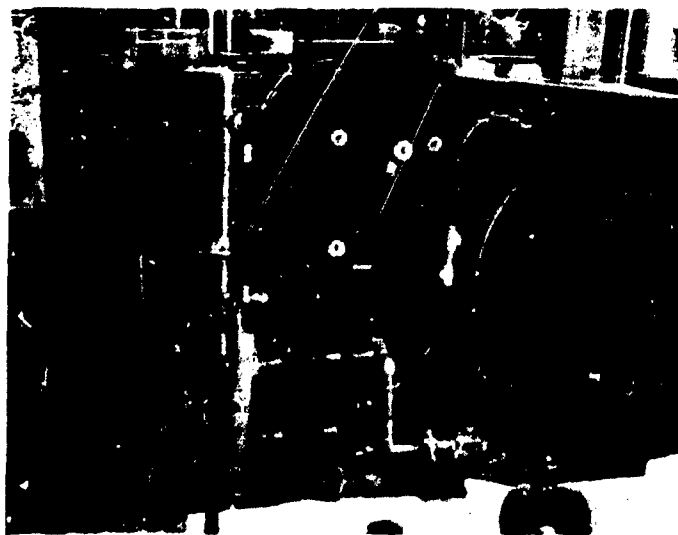


Fig. 21. Hydraulic system of electrodialysis unit.

F9353

was that the stack contained 96 cell-pairs (48 cell-pairs in each half of the stack) instead of 100 cell-pairs. The two extra sets of holes found in the old-type spacers seemed to be of no significant use, and, therefore, new spacers with only two sets of holes were used. Previous tests at Fort Belvoir showed that the old-type (four sets of holes) spacer was unsatisfactory (Fig. 12).

For the decontamination tests, a 3,000-gallon feed-water tank containing water from well J-11 was used. A solution containing 144 millicuries of Cs^{137} in the chloride form was added to the well water to an initial level of $1.26 \times 10^{-2} \mu\text{c/ml}$.

b. Tests and Test Results.

(1) Demineralization Studies.

(a) Run Number 1. This test consisted of the demineralization of a brackish well water containing 889 mg/l TDS to a level of 500 mg/l or below. This initial test started on 30 September 1959 and lasted until 22 October 1959. The total operating time was 100 hours.

Following is an analysis of brackish water from well J-11:

	Concentration mg/l
Calcium, Ca	82.4
Magnesium, Mg	12.4
Iron, Fe	1.3
Chloride, Cl	18.1
Bicarbonate, HCO_3	53.7
Silica, SiO_2 (non-ionic)	(64)
Sulfate, SO_4	528
Total Dissolved Solids	889
pH	7.8 units

On 24 September, the electrodialysis unit was assembled inside the building. New perm-selective membranes and membrane spacers (Figs. 22 and 23) were used as components in the stack. Brackish water was pumped to the unit from the 500-gallon tank outside the building.

From 25 to 26 September, the unit was tested for leaks and other hydraulic and mechanical faults. The membrane stack showed no excessive leakage such as that experienced in the Fort Belvoir tests. The conductivity control was recalibrated, and it was determined that a meter



Fig. 22. Membrane stack.

F11119



Fig. 23. Membrane stack disassembled (acid storage tank shown to rear of stack).

F11114

setting of 300 μ mhos/cm would correspond to an effluent product concentration of approximately 300 mg/l TDS.

The initial run began on 30 September, and no appreciable problems were encountered until 8 October. The stack was electrically probed with a portable ohmmeter to check the membrane stack for high-resistance spots because the production rate had decreased from 63.4 to 42.5 gph during that period. Table IV includes operating data for the period 30 September through 8 October 1958.

Table IV. Operating Data, 30 September - 8 October^(a)

Date	T _F	V _s	C _s	P _a	F	R _p	B _w	E _w	P _f	A	t _o
30 Sep	75	56	0.8	63.4	0.6	3.7	12.4	26.3	1.9	28	7.1
30 Sep-1 Oct	74	57	0.7	55.0	0.6	3.8	13.4	26.3	3.0	28	14.3
2 Oct	74	57	0.6	51.7	0.6	3.8	13.0	25.4	3.9	28	3.1
2-3 Oct	72	56	0.6	47.7	0.6	3.8	17.2	24.6	5.0 ^(b)	28	14.8
5 Oct	70	56	0.55	42.2	0.6	3.8	-	24.9	1.6	35	5.8
6 Oct	75	57.5	0.5	44.7	0.6	3.8	20.2	24.6	2.4	35	6.9
7 Oct	72	57.5	0.5	40.2	0.5	3.8	16.6	24.2	2.5	35	7.9
8 Oct	75	56	0.42	42.4	0.6	3.7	18.6	25.2	4.9	35	7.5

- (a) Where: T_F = average temperature of feed water, °F.
V_s = voltage at start of cycle, volts.
C_s = current at start of cycle, amps.
P_a = average reproduction rate, gph.
F = feed water to brine recirculation, gpm.
R_p = product recirculation, gpm.
B_w = brine waste stream rate, gph.
E_w = electrode waste stream rate, gph.
P_f = pressure drop across filters, psi.
A = acid (1-2% NaHSO₄) feed to concentration and electrode cells, gallons per 24-hour day.
t_o = operating time, hours.

- (b) Filter cartridges in feed line replaced.

The data included in Table IV show a marked decrease in production rate during the first 67 hours of operation. Also at the start of the cycle, the progressive drop in current and the presence of a particularly high

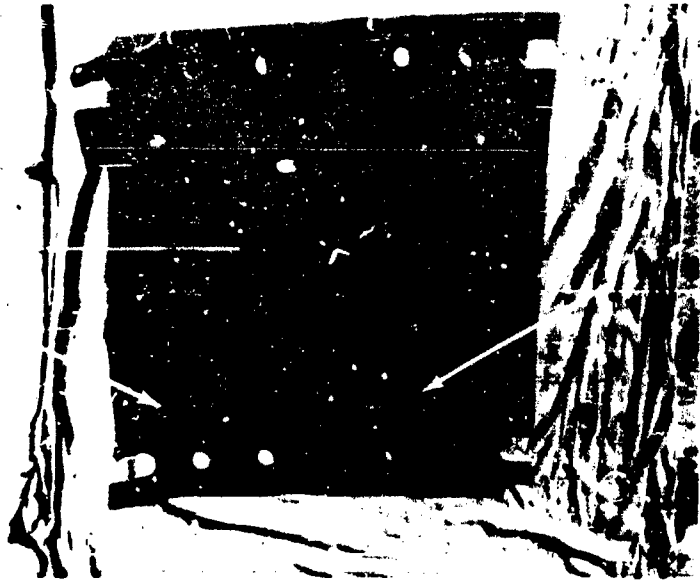


Fig. 24. Anion membrane depicting iron slime (see arrows).

F9420

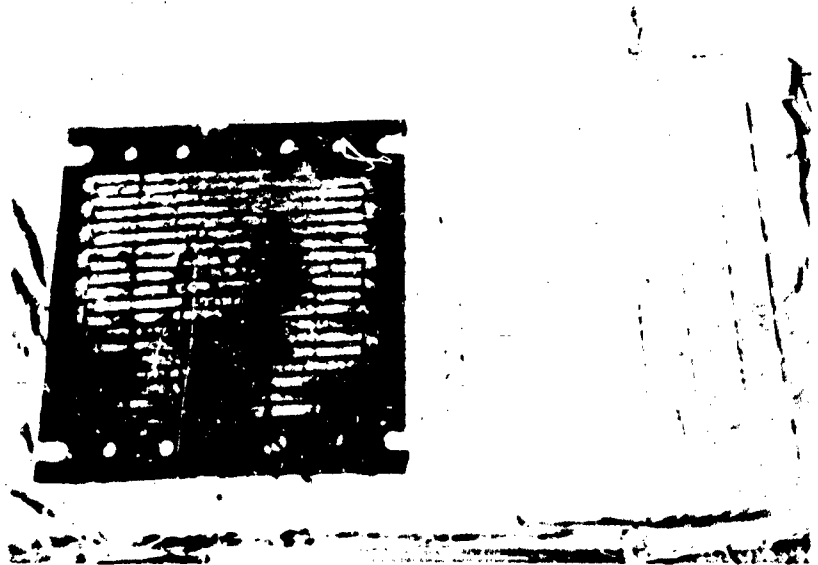


Fig. 25. Precipitated silica on anion membrane and spacer.

F9419

resistance spot approximately 1.5 inches below the bottom anode indicated that there was a stoppage in flow between one or more cells. There were other resistance spots through the entire membrane stack but none as high as the one previously mentioned.

Upon inspection of the stack components, it was found that practically all of the anion membranes were covered with a thin, brown slime which was analyzed and found to contain iron. Some anion membranes were covered with more slime than others, but the slime was invariably found on the dilution side of the anion membranes. In Fig. 24, the arrows indicate the iron slime deposits, particularly on the bottom section of the membrane (the lighter sections resembling flow paths contain the iron slime). The anion membranes and spacers were soaked in 0.5 N HCl for approximately 24 hours. This acid cleaning removed the last traces of slime from the membranes and spacers. The 500-gallon feed tank outside of the shack was refilled with brackish water. Six pounds of citric acid was added to this tank, and the 500 gallons of water was pumped through the unit (rectifier off) to flush the hydraulic system of all iron deposits. This water was pumped to the unit at a rate of 1.2 gpm.

When the lower half of the stack was inspected closer on 19 October, it was found that one entire cell was completely blocked with silica scale on the concentration side of the anion membrane (Fig. 25). This silica was a hard, solid material which precipitated in that particular cell because a spacer was incorrectly assembled. Blockage of the cell was complete; no water flow was allowed through the cell. The anion membrane had to be replaced since the silica deposit could not be removed.

Figure 26 shows the decrease in production rate (during the first 67 hours of the run) due to iron sliming. After the anion membranes were acid cleaned and the unit was flushed, the production rate increased but dropped off rapidly because of the blocked cell caused by the silica deposits. After four anion membranes were replaced because of the silica deposits, the production increased rapidly to a 70-gph rate.

The experience gained from this first test led to the conclusion that proper pH control of the brine blowdown stream would prevent iron sliming and silica scaling of the membranes.

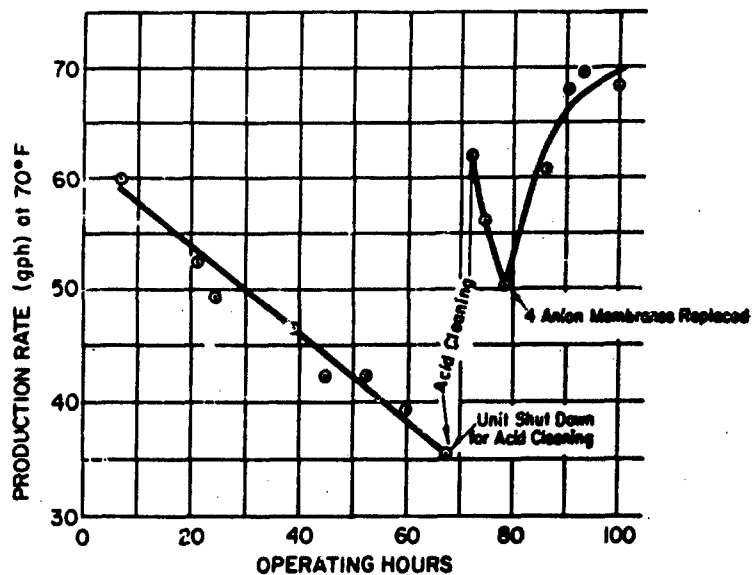


Fig. 26. Effects of iron sliming and acid cleaning on production rate.

The data included in Table V represent effectiveness of the electrodialytic process for removal of ionized salts from brackish waters.

Table V. Removal of Ionized Salts

Ion	Raw Water (mg/l)	Product (mg/l)	Removal (%)
Ca	82.4	19	77
Mg	12.4	3.2	74
Fe	1.3	0.6	54
Cl	18.1	7	61
HCO ₃	53.7	26	52
SO ₄	528.0	134	75
SiO ₂ (non-ionic)	(64.)	(64)	0
TDS	889.0	311	65

The data in Table V are for the 100-hour run. Concentrations of dissolved salts in the product stream, as shown

above, are average values taken during the first run. Silica was not removed. Silica existed, not in the ionized form, but as a colloidal, unreactive substance with no ionic charge and, therefore, was not removed by the electrodialysis unit.⁴

(b) Run Number 2. On 28 October, brackish water having the following analysis was pumped to the electrodialysis unit:

	<u>Concentration, mg/l</u>
Calcium, Ca	84.0
Magnesium, Mg	10.2
Iron, Fe	0.8
Chloride, Cl	18.5
Sulfate, SO ₄	522.
Bicarbonate, HCO ₃	53.6
Silica, SiO ₂	(64) Non-ionized, colloidal
Total Dissolved Solids	788
pH	7.8 units

It was found that a setting of 20 μ mhos/cm on the conductivity meter would give an effluent product with a concentration of 5 mg/l TDS as NaCl.⁵ A setting of 0 μ mhos/cm corresponded to a product concentration of 1.5 mg/l TDS as NaCl. The feed water also contained 1.26×10^{-2} μ curie/ml Cs¹³⁷ in the chloride form, but the study on radioactivity removal will be discussed later in the report.

Table VI includes operating data for the period 28 October to 3 November.

On 29 October, water samples of the product and brine stream were taken at precise time intervals during the fourth cycle. (A cycle is the time required to demineralize one batch of water or about 25 gallons.) Total chemical analyses were made on each individual sample to determine the total dissolved solids content. Accurate recordings of the stack current were made with a special ammeter which could be read to plus or minus 0.005 amp. The results are shown in Fig. 27. Since the current

4. This assumption was used in the calculation of total dissolved solids in Test No. 2.

5. A special purity meter was used.

Table VI. Operating Data, 28 October - 3 November*

Date	T _F	V _S	V _E	C _S	C _E	P _A	F	R _P	B _W	E _W	A	t _O	S
28 Oct	68	56	58	0.65	0.033	14.6	0.6	3.7	21.5	21.4	2	7.0	5.0
29 Oct	59	56	58	0.58	0.032	12.8	0.6	3.6	23.4	22.0	2	7.3	5.0
30 Oct	50	56	58	0.58	0.030	9.9	0.6	3.5	18.0	23.8	2	6.8	5.0
31 Oct	51	56	58	0.52	0.030	9.7	0.6	3.5	19.8	23.4	2	7.0	4.8
2 Nov	52	54.5	56	0.54	0.018	6.1	0.6	3.5	20.2	22.5	2	7.4	1.5
3 Nov	51	56.2	57.8	0.52	0.018	5.9	0.6	3.5	19.6	23.0	2	3.8	1.5

* Where: T_F = average temperature of feed water, °F.

V_S = voltage at start of cycle, volts.

V_E = voltage at end of cycle, volts.

C_S = current at start of cycle, amps.

C_E = current at end of cycle, amps.

P_A = average production rate, gph.

F = feed water to brine recirculation, gpm.

R_P = product recirculation, gpm.

B_W = brine waste stream rate, gph.

E_W = electrode waste stream rate, gph.

A = acid (1.5% NaHSO₄) feed, gph.

t_O = operating time, hours.

S = product concentration, mg/l TDS as NaCl.

is proportional to the total dissolved solids content of the water being demineralized, one would expect the curves to be alike. The cycle time was 98.7 minutes. The production rate at 59° F was 12.8 gph. About 91 percent of the total dissolved solids was removed after 40 minutes of operation; 96 percent removal was accomplished after 60 minutes of operation. The concentration of the brine stream decreased with time due to the fact that more minerals or ions were removed from the feed water in the beginning or early part of the cycle than at the end.

Since the demineralization unit operates on a batch cycle, a specific quantity of raw water is recirculated, and an approximate 40-percent reduction in total dissolved solids is realized. Each recirculation of this specific quantity of water is known as a "pass." In order to demineralize a brackish water containing 800 mg/l TDS down to

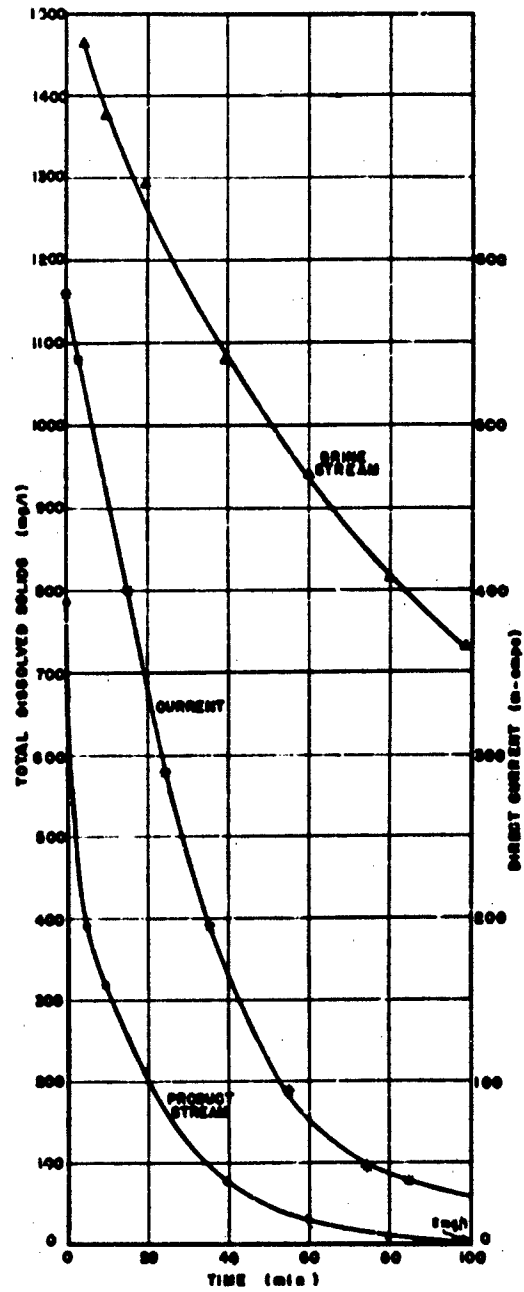


Fig. 27. Characteristics of a typical demineralization cycle.

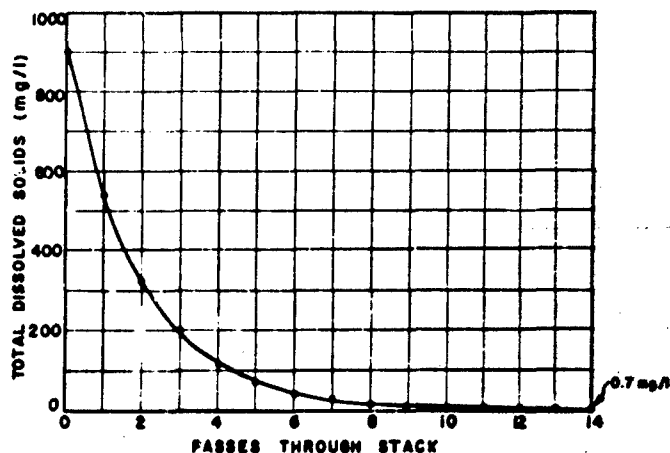


Fig. 28. Total dissolved solids removal per pass through stack.

300 mg/l TDS, approximately three passes through the stack are required (Fig. 28). It takes approximately ten passes through the membrane stack to demineralize the same water down to a level of 5 mg/l TDS. It was found experimentally that each pass took about 10 minutes. From these facts, the production rate can be approximated for any given set of conditions.

Table VII represents the results of the demineralization tests which were conducted in Jackass Flats, Nevada.

Table VII. Demineralization Test Results

Characteristic	Test Number		
	1	2	3
Feed water, mg/l TDS	889	788	788
Product water, mg/l TDS	311	5.0*	1.5*
Product water, gal.	5520	332	67
Brine Waste, gal.	1720	583	244
Electrode waste, gal.	2360	635	253
Total water used, gal.	9600	1552	544
Length of test, hrs	100.4	28.1	11.2
A.C. power used (total KW-HR)	66	17	6
D.C. to stack, KW-HR	5.05	0.42	0.10
Average temp., °F	74	57	51
Power consumption, KW-HR/1000 gal.	12	52	90

* mg/l TDS as NaCl

The power consumption figures were calculated at the average temperatures given. The relationship between the amount of total dissolved solids removed from the feed-water stream and the power consumed during the demineralization is expressed in Fig. 29. Figure 29 shows that the power consumption increased moderately as the feed water was being demineralized, but there was a marked increase in the power consumption after 89 percent (800 mg/l) of the ionized salts was removed.

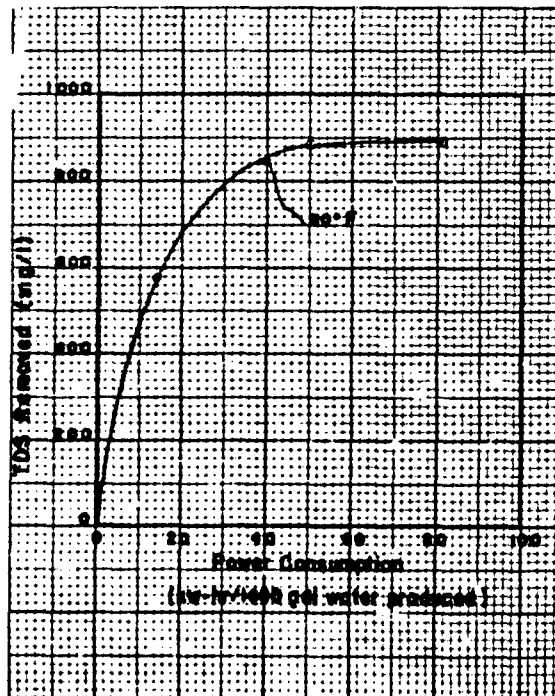


Fig. 29. Effect of TDS removal on power consumption.

(2) Decontamination Studies. On 28 October, raw water containing 1.26×10^{-2} μ curies/ml cesium-137 was fed to the electrodialysis unit. The conductivity control setting was 20 μ mhos/cm. Film badges were placed on the unit in relevant positions in order to determine the radioactivity pickup by the unit at various locations. Figure 30 shows the positions of the film badges, and the amount of radioactivity accumulated during the tests is expressed in Table VIII. Film badges were placed on the unit in the following positions:

Fig. 30. Positions of film badges on unit (positions described in Table VIII). F10372

- (a) Inlet feed-water line.
- (b) Product recirculation line ahead of the filter.
- (c) Discharge side of the brine recirculation pump prior to the stack.
- (d) Brine discharge line to waste.
- (e) Electrode waste discharge line.
- (f) Product water discharge line.
- (g) Upper half of the stack.
- (h) Lower half of the stack.

Table VIII. Film Badge Data

Number	Film Badge Positions*	Total Time (hr)	Total (mrem)	Average (mrem/hr)
1	Inlet Raw Water Line	147.3 ^(a)	475	3.22
2	Product Recirculation Line	147.3	130	0.88
3	Brine Discharge Line from Pump to Unit	147.3	140	0.95
4	Brine Discharge Line to Waste	147.3	300	2.04
5	Electrode Line Discharge	147.3	220	1.49
6	Product Discharge Line	147.3	110	0.75
7	Top Stack, Facing	142.5 ^(b)	2900	20.3
8	Bottom Stack, Facing	142.5	2900	20.3

Film badges attached 28 October 1959; (a) at 1415 hours, (b) at 0930 hours; removed 3 November 1959 at 1245 hours.

<u>*Personnel</u>	<u>Film Badge Reading</u> (mrem)
Experimenter	10
Operator No. 1	10
Operator No. 2	<10

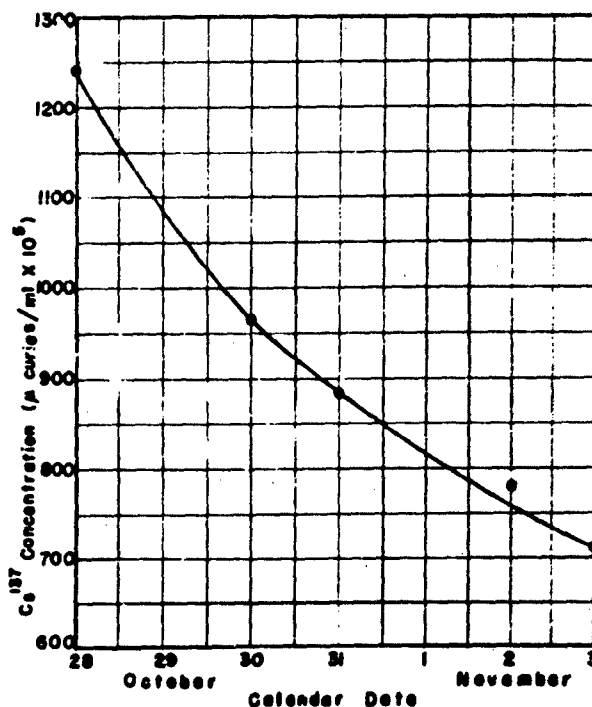


Fig. 31. Cesium-137 concentration decrease in feed tank (at point of exit) during decontamination tests.

On 2 November, the conductivity setting was changed to 0 μ mhos/cm. The concentration of the feed water was now 0.81×10^{-2} μ curies/ml or a 36-percent decrease from the initial contamination. Figure 31 shows this decrease in feed water contamination. Since there were many dust storms in Jackass Flats, Nevada, and the 3,000-gallon raw water tank was not covered, it may be assumed that sand and dust particles entered the tank. When this foreign, inert material settled, it is probable that the cesium-137 was adsorbed on these particles and carried down to the bottom of the tank.

On 29 October, water samples were simultaneously taken of both the product and brine streams during cycle No. 4. The reason for the sampling was to determine the rate of removal of cesium-137 radioactivity from the feed water and the rate of concentration of this radioactivity in the brine blowdown stream (Fig. 32). Approximately 85 percent of the cesium-137 radioactivity had been removed after 20 minutes of operation.

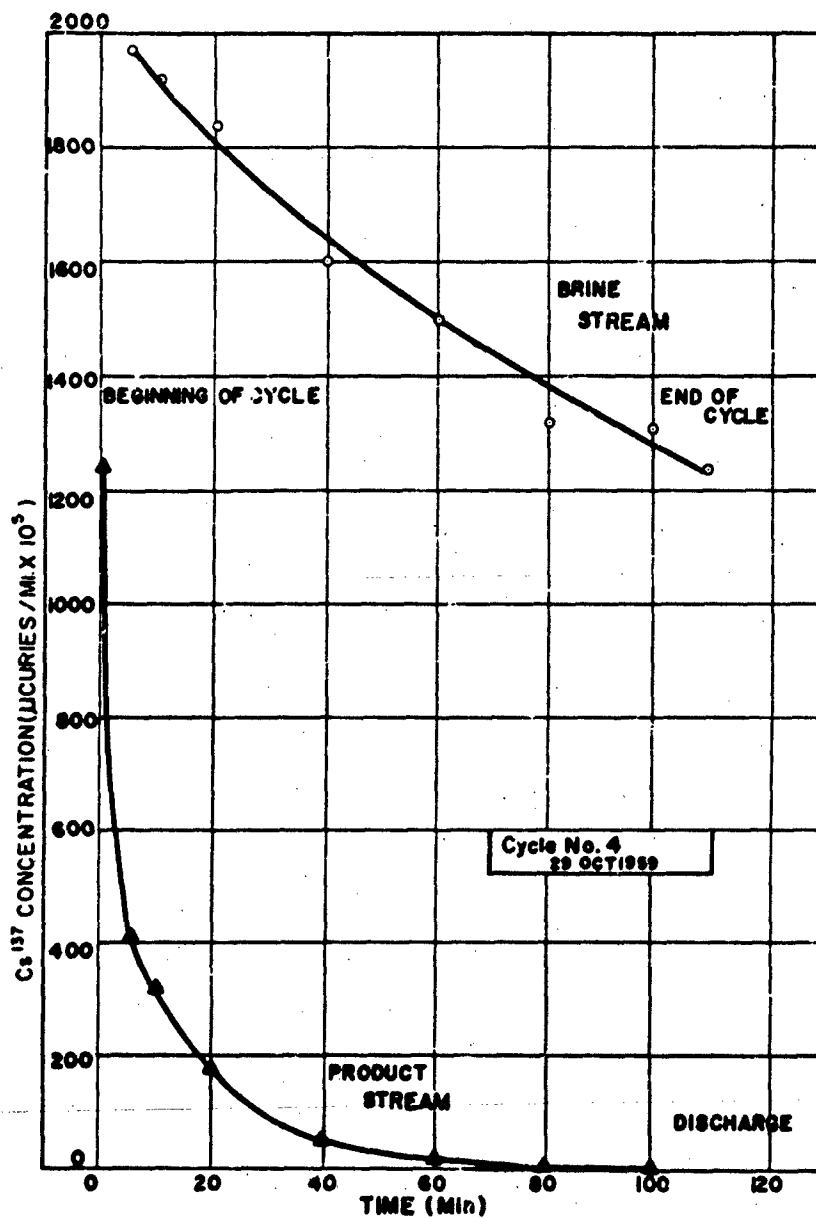


Fig. 32. Radioactivity removal by unit.

The length of the cycle was 98.7 minutes. Over 99.9 percent removal was realized.

The results of the decontamination studies utilizing the electrodialysis unit were as shown in Table IX. The total operating time of 39.3 hours with 99.4 to 99.9 percent of the initial radioactivity removed by the electrodialysis unit.

Table IX. Results of Decontamination Tests
Conducted in Jackass Flats

Date	Cond. Setting (a)	Raw ater (b)	Product (b)	Brine Waste (b)	Elec- trode (b)	% Removal
28 Oct	20	1.24×10^{-2}	18×10^{-6}	1.33×10^{-2}	1.29×10^{-2}	99.9
29 Oct	20	1.1	24	1.29	1.31	99.8
30 Oct	20	0.97	56	0.98	0.98	99.4
31 Oct	20	0.88	48	0.88	0.82	99.5
2 Nov	0	0.81	9	0.78	0.74	99.9
3 Nov	0	0.71	12	0.71	0.66	99.8

(a) Expressed as μ mhos/cm.

(b) Cesium-137 concentration, μ curies/ml.

The electrodialysis unit was lecontaminated upon completion of the decontamination studies. Approximately 5 pounds of citric acid was dissolved in the 500-gallon feed tank which contained brackish water from well J-11. This acidified water ($\text{pH} \leq 3$) was fed to the unit at a rate of 1.0 gpm. The exterior of the membrane stack, before decontamination, gave a reading of 12 mr/hr on the survey meter. After the unit was acid flushed for 4 hours (rectifier off), the survey meter indicated 7 mr/hr about the membrane stack. The unit was again flushed with raw water (no acid) for approximately 4 hours. Survey meter readings of various sections of the unit were less than 1 mr/hr. These readings were of the hydraulic system only; not the membrane stack. The membranes were removed from the stack and placed in two 15-gallon polyethylene drums containing a weak citric acid solution for approximately 1 month after which a reading of less than 3 mr/hr was obtained.

III. DISCUSSION

7. Examination of Test Results. A series of tests was conducted on the newly acquired electric-membrane demineralizer. The purpose of these tests was to obtain operational data of the process

for the demineralization of water. In the early tests, the demineralizer was operated to obtain information relative to the operating procedures involved. In addition to the test data presented in sections II and III of this report, Appendix B includes all tabulated data with reference to the graphs used and all chemical analyses performed during the tests.

During the initial tests in 1958, sliming of the membranes due to iron caused many difficulties. This sliming resulted in an increase in membrane electrical resistance and a decrease in production rate. In one instance, the pressure drop across the inlet filters was noted to be 15 psi. After the unit was shut down, a careful inspection of the filter was made. It was found that the two filter cartridges were completely plugged with a brown, mud-like substance. It was later determined that the maximum safe pressure drop across the inlet filters was 5.0 psi.

Clarification of the feed water prior to demineralization was necessary. If not eliminated, the turbidity and suspended matter entering the unit will be adsorbed on the membrane surfaces blocking membrane "pores" and increasing membrane resistance. This will prevent the dialysis process from functioning properly. If the pressure drop across the filters exceeds a maximum value, turbidity penetrates the filter and is discharged into the feed-water stream to the membranes.

The brown, mud-like substance mentioned above was analyzed qualitatively and found to contain appreciable amounts of iron. Four hundred and fifty gallons of 0.03 percent citric acid containing 2,830 mg/l NaCl (pH - 3.3) was fed to the unit to flush the hydraulic system. This removed the iron slime and any acid-soluble scale (CaCO_3 , $\text{Mg}(\text{OH})_2$, etc.) formed. Citric acid is a very useful solid acid. When dissolved in water and fed to the membrane stack, citric acid acts as an acid to neutralize any carbonates, hydroxides, or oxides formed and as a chelating agent to sequester magnesium and calcium ions which may form scale. In some cases, the acid flushing did not remove all of the iron slime from the membranes. The membranes had to be scrubbed individually to effect an efficient removal of the slime. If the feed water contains an appreciable concentration of iron, particularly in the ferrous state, pretreatment by aeration or coagulation and filtration may be necessary. If iron is allowed to enter the membrane stack untreated, the anion membranes will be impaired. In all cases, the iron slime was found on the dilution side of the anion membranes. The iron in the feed water will react with the hydroxide formed as a result of the alkalinity present (at pH \geq 7.0) and form hydroxides of iron (ferric or ferrous). It was not these specific hydroxides which caused the sliming, however, or the iron slime would have been found on the dilute side of the cation membrane. Iron slime was never found on this membrane.

(The assumption is made that concentration polarization conditions did not exist at any time.) It was an anionic form of iron hydroxide, $\text{Fe}(\text{OH})_3$, which is believed to have been the direct cause of the blockage. The $\text{Fe}(\text{OH})_3$ would be attracted to the anode, but since it is a relatively large anion it is not able to pass through the anion membrane "pores" and thus would begin to build up on the dilute side of the membrane, increasing its electrical resistance (Fig. 24).

The high-resistance spots in the membrane stack were detected with the use of a portable volt-ohmmeter. The sections of high resistance were found by probing; when the high-resistance spot was found, the meter showed a definite increase in voltage.

The iron hydroxides which were not adsorbed on the anion membranes were removed by a cartridge-type filter placed in the product recirculation line. Much brown-like material was removed by this filter during both the Fort Belvoir tests and the field studies at Nevada. It is believed that filtration of recirculated water is helpful and will extend maintenance-free operating cycles.

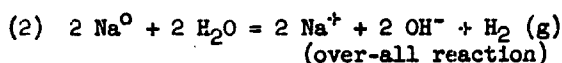
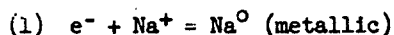
The brackish water used at Jackass Flats, Nevada, contained some 64 mg/l silica. Results indicate that the electrodialysis unit did not remove silica. In one instance, electrical probing of the stack disclosed an extremely high resistance in the bottom section of the membrane stack. The cause of the high resistance was apparent when the stack was taken apart. One complete cell was filled with precipitated silica on the concentrate side of the anion, while on the dilute side iron slime was found (Fig. 25). The cell was so filled with silica that there was no water flow through the cell. The stoppage was caused by a spacer which had been incorrectly fabricated. The anion membrane and spacer had to be replaced, since the silica could not be removed without the anion membrane being damaged. It was found that if the pH of the brine stream were kept high ($\text{pH} > 7.0$) in order to prevent silica scaling, iron hydroxide would begin to precipitate in the cells and sliming would result; however, if the pH of the brine stream were lowered to 3.0, silicic acid, $\text{H}_2\text{SiO}_3 \cdot \text{xH}_2\text{O}$, would form on the membranes. The pH of the brine stream was, therefore, maintained between 6.5 to 7.5 without further scaling or sliming difficulties during the remainder of the field tests.

During the joint decontamination-demeralization tests with the electrodialysis unit, an unusual problem appeared. With a setting of 0 μ mhos/cm on the conductivity meter, an effluent product water containing 1.5 mg/l TDS as NaCl resulted. This measurement was conducted using a purity meter specifically designed for low salt concentrations (less than 15.0 mg/l TDS as NaCl). A silica determination was done on this same product water sample; the sample was found to contain about 64 mg/l SiO_2 . From this fact, it was

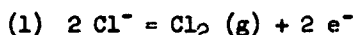
concluded that silica was not removed by the electrodialysis process and existed as a non-ionic, unreactive substance.

Figure 1 depicts a cross-sectional side view of the membrane stack. It can be seen that the electrode (anode and cathode) cell effluents are discharged to waste. Since chlorine, hydrogen, and oxygen gases are evolved at these electrodes, the electrode cells must be flushed with water. This effluent waste stream must then be suitably vented and discharged. The following chemical equations are the reactions which occur at the electrodes:

a. Cathode, negative electrode.



b. Anode, positive electrode.



It can be seen from equation a(2) that hydroxide ions are liberated at the cathodes. The concentration of hydroxide ion is proportional to the current in the stack; since the current is at its peak in the commencement of a cycle, the hydroxide-ion concentration reaches a maximum then also (Fig. 16). It is necessary, therefore, to neutralize the hydroxide ions formed with acid or scale will build up in the electrode cells. The pH of this electrode stream must not exceed 4.0 at any time. The acid concentration and rate of feed to the electrode stream are dependent upon peak d-c voltage assuming, of course, that the acid is mixed with demineralized water to obtain the desired initial concentration.

The same is also true of the brine blowdown stream. Acid must be fed to this waste stream in order to prevent the formation of calcium carbonate scale. The concentration and acid feed rate is dependent upon the total dissolved solids, pH, calcium, and bicarbonate concentration of the brine waste stream and its temperature. From these data, a Langelier Index is calculated (example given in Fig. 33). A Langelier, or scaling, Index is equal to the actual pH minus the sum pH obtained from the chart. Any positive index obtained indicates a scaling condition; acid, therefore, must be added to the brine waste stream to remove bicarbonate ion, HCO_3^- , until an index of less than -0.5 is obtained. As can be seen in the example given, the index is -0.96, a negative index, which indicates the existence of a safe operating condition.

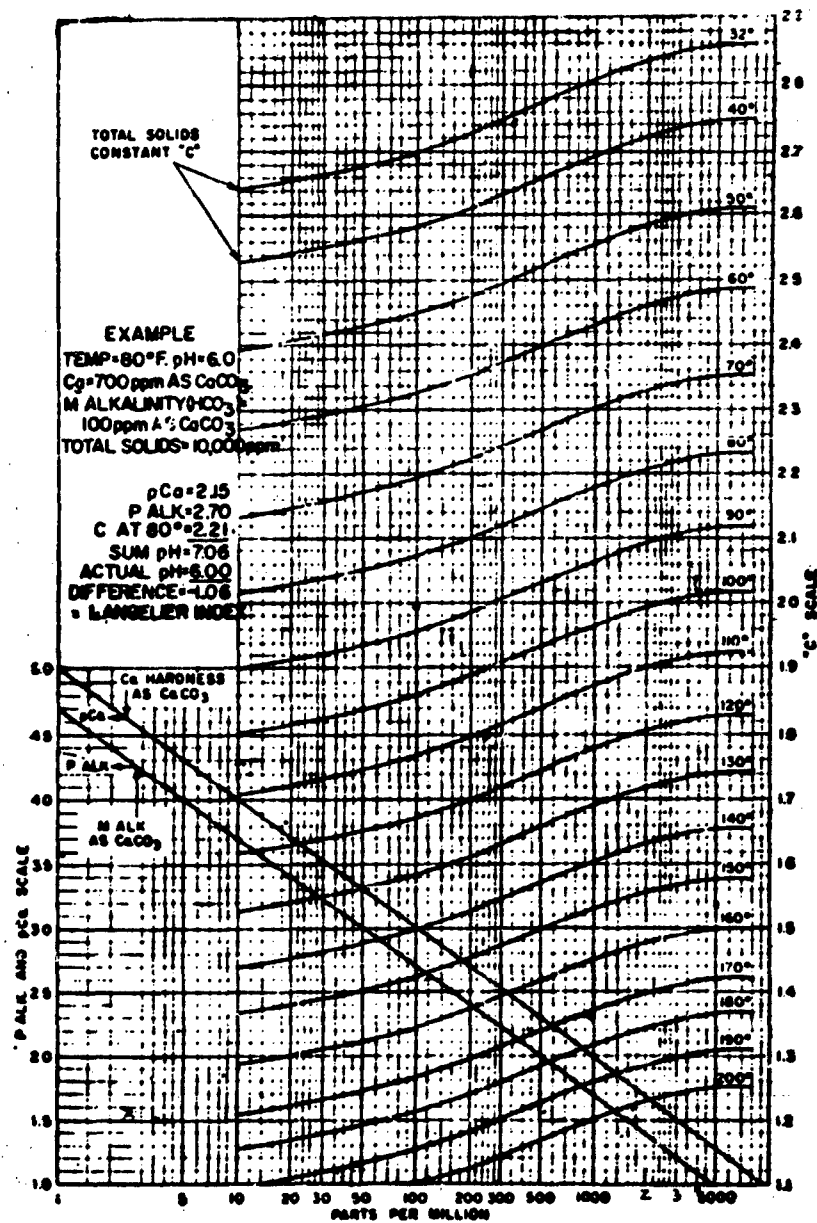


Fig. 33. Langelier saturation-index chart.

Because of problems involving transportation and handling, a solid acid salt such as sodium bisulfate, NaHSO_4 , was used. When mixed with deionized water, sodium bisulfate becomes an acid by ionization. The acid solution of bisulfate is injected from a polyethylene-lined drum into both the electrode and brine blowdown streams by a corrosion-resistant duplex proportioning pump. The acid feed rate to each stream is controlled by adjustment of the acid-pump strokes. If scaling conditions exist (positive Langelier Index), the acid is fed accordingly to the brine waste stream as determined by the Langelier Index. Bisulfate concentrations between 1 and 2 percent were used in Nevada; the reason for these low acid concentration was the low alkalinity (54 mg/l as HCO_3) present in the brackish water. Citric acid may be used to decontaminate and also to remove soluble precipitates such as CaCO_3 and $\text{Mg}(\text{OH})_2$.

Since the brackish water from well J-11 in Jackass Flats contained approximately 82 mg/l calcium ion and 525 mg/l sulfate ion, the brine waste stream had to be analyzed frequently in the early stages of the field tests to determine if the calcium sulfate concentration was reaching or would exceed the theoretical solubility limit. The feed water to the brine blowdown stream was kept at 0.6 gpm in order to prevent calcium sulfate precipitation and silica scale formation in the membrane compartments.⁶

The decontamination and demineralization tests were performed simultaneously on brackish water containing 788 mg/l TDS and 1.26×10^{-2} μ curies/ml cesium-137 dissolved radioactivity. The results of the chemical analyses of water samples collected during the test are plotted in Fig. 34.

The drinking water tolerance for one year of military consumption is 3×10^{-4} curies/ml. After 50 minutes of operation during cycle No. 4, the cesium-137 dissolved radioactivity was removed by the electrodialysis unit to approximately 3×10^{-4} μ curies/ml; at this time, there remained only about 50 mg/l TDS in the product stream; this was a 94-percent removal of total dissolved solids. A production rate of approximately 33 gph at 70° F would be realized if the cycle time were cut from 98.7 to 50 minutes and the effluent product would be within the tolerance level of 3×10^{-4} μ curies/ml and a total dissolved solids content of 50 mg/l. From the previous tests, it was concluded that the electrodialysis process was capable of removing ionized salts from brackish waters down to a level of 1.5 mg/l TDS as NaCl .

Figures 35 through 38 depict the removal of specific ions during a typical demineralization cycle (800 mg/l TDS to 5 mg/l TDS).

6. Further information on the scaling problem is included in reference 13.

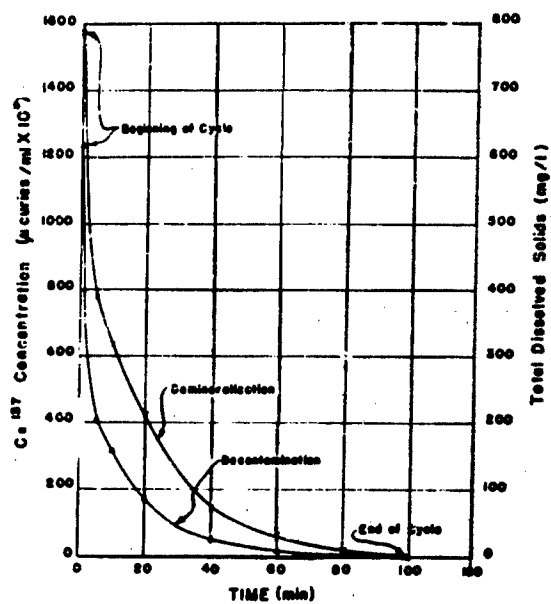


Fig. 34. Reduction in total dissolved solids and cesium-137 radioactivity during one complete electro dialysis cycle.

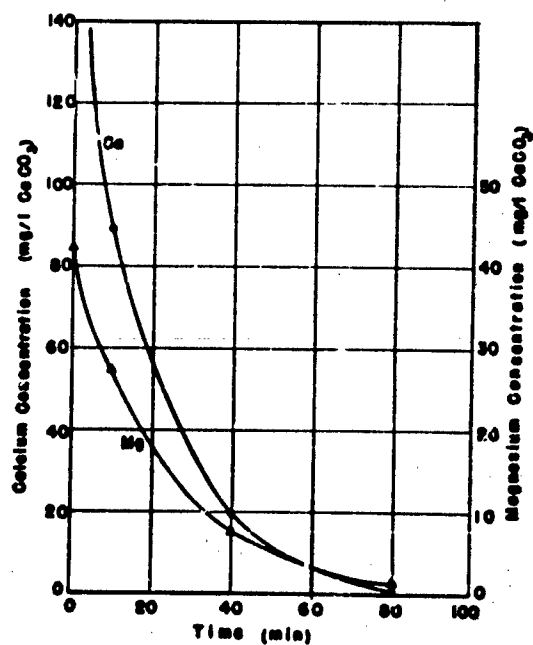


Fig. 35. Rate of hardness removal.

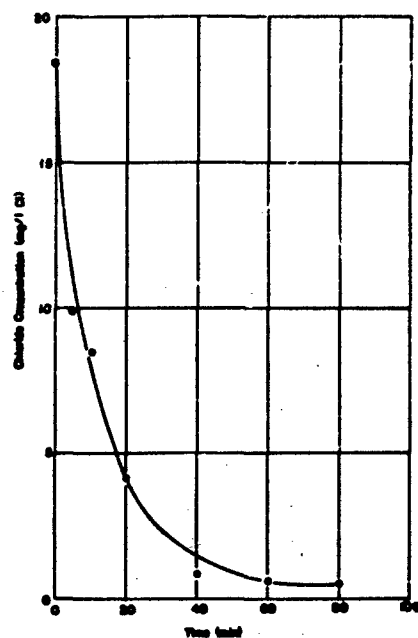


Fig. 36. Rate of chloride removal.

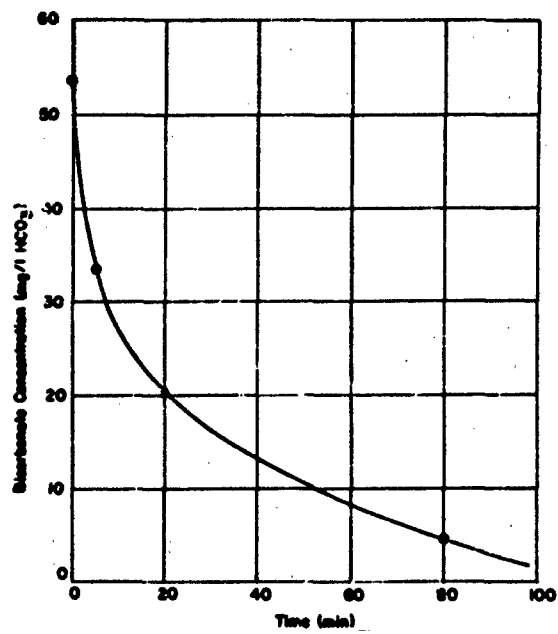


Fig. 37. Rate of alkalinity removal (HCO₃⁻).

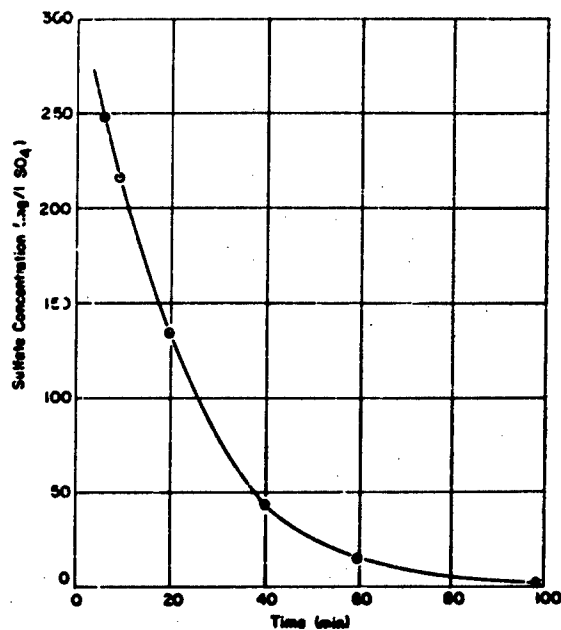


Fig. 38. Rate of sulfate removal.

Table X, based on data taken from Figs. 35 through 38, shows that after 60 minutes of operation calcium and magnesium ions were removed to approximately the same extent; sulfate and chloride ions were also removed in like manner and bicarbonate ion showed a removal of 84 percent after 60 minutes of operation.

Table X. Demineralization Results

Cycle No. 4 After 60-Minute Operation	Initial Concentration (mg/l)	Product Concentration (mg/l)	Removal (%)
Ca, as CaCO ₃	210	7.7	99
Mg, as CaCO ₃	42	1.8	95
SO ₄	522	15	97
Cl	18.5	0.7	96
HCO ₃	53.6	8.7	84

On 19 September 1958, the electrodialysis unit was shut down due to unusually high resistance in the membrane stack. Upon further inspection of the stack, many spacers were found with sections literally "melted." The melted sections were opaque, yellow in color. The spacers, composed of a polyvinylchloride-type plastic material, showed appreciable damage around the "holes" (Fig. 12). The adjacent membranes appeared to be burned and were warped in similar places, so much so as to render them incapable of performing efficiently; the damaged membranes, therefore, had to be replaced. In the Nevada field tests, a new type of spacer was used: one having only two sets of holes, instead of the old type containing four sets of holes (Fig. 12). These new spacers performed exceptionally well in the field. It is believed that the reason for the melting at the holes of the old type of spacer was due to degradation of the polyvinylchloride plastic material. This degradation was caused by chlorine or chloride ions retained in the holes of the spacers (due to high current densities in these areas) during demineralization.

The only other difficulty which involved a membrane spacer occurred during the Nevada field tests. A spacer which was not properly fabricated caused a cell in the membrane stack to become filled with silica deposits. After this spacer was replaced with a new one, no more scaling or blockage problems were encountered during the tests.

The film badge data (Table VIII) indicate that the maximum cesium-137 radioactivity is emitted from the membrane stack during operation (2,900 mrem total). The average dosage from the stack was 20.3 mrem/hr during the decontamination studies run of approximately 39 hours. This dosage is relatively low and would not present a hazard to the operator. When the film badges worn by the experimenter and the operators were developed, cumulative dosages of 10 mrem were recorded in all cases. Since the dosimeters or film badges used give cumulative recordings, the dosage may eventually become relatively high and may present a hazard to the operator. The membranes may have to be decontaminated with acid to remove this absorbed radioactivity.

Decontamination of the electrodialysis unit was accomplished during the period 4 to 6 November. Five hundred gallons of 0.15 percent citric acid was pumped to the unit at a rate of 1.0 gpm. A beta-gamma survey meter was used to probe possible hot-spots in the unit. The maximum reading, 12 mr/hr, occurred at the exterior of the membrane stack before decontamination with acid; the reading was reduced to 7 mr/hr after 4 hours of acid flushing. Five hundred gallons of raw brackish water (no acid) was pumped to the unit to remove the last traces of acid. The maximum reading found when the unit was probed was 2 mr/hr. The membrane stack was stored in two 15-gallon polyethylene drums containing a weak citric acid solution.

The membranes, 4 weeks later, gave a reading of less than 1 mr/hr after they were flushed repeatedly with tap water.

8. Evaluation of the Unit. Laboratory and field tests showed that the electrodialysis process is capable of removing ionized salts from brackish waters containing up to 12,000 mg/l TDS; however, the higher the concentration of the feed water to be demineralized, the higher the power consumption and the lower the production rate. The feed water temperature also affects the production rate; each degree Fahrenheit rise in feed water temperature results in a 1.1 percent rise in production rate.

The results of the decontamination studies conducted in the field indicated a removal of more than 99 percent of the dissolved radioisotope cesium-137. When developed, film badges placed in strategic positions on the unit showed a maximum average dosage of 20.3 mrem/hr about the membrane stack itself. The unit can be effectively decontaminated with a dilute citric acid solution.

a. Operator Techniques. The operator should have a working knowledge of water treatment such as that taught to a water supply specialist. Some techniques that the operator in the field will be required to be familiar with include:

- (1) How to assemble the membrane stack properly.
- (2) How to perform chemical tests on the feed water, and based on results change operating procedures to obtain best performance.
- (3) How to adjust the brine blowdown stream flow rate for high sulfate concentrations in order to prevent calcium sulfate scale formation in the membrane cells.
- (4) How to determine concentration and flow rate of acid necessary to reduce alkalinity in the brine waste stream and hydroxide ion concentration in the electrode waste stream.

b. Operational Requirements. The following are operational requirements for an electrodialysis unit:

- (1) A chemical analysis of the feed water is necessary to determine calcium and magnesium hardness, alkalinity, iron, sulfate, chloride, pH, turbidity and/or silica, and, if necessary, sulfide and manganese. A compact water analysis kit can be developed for field operation.
- (2) Iron oxides, turbidity, slime, algae, and bacteria must be removed from the feed water by suitable pretreatment

methods before being pumped to the membrane stack for demineralization.

(3) The pH of the brine blowdown stream (by constant acid feeding) should be kept from 0.5 to 1.0 pH unit below the pH of the feed water in order to minimize the calcium carbonate scaling potential.

(4) Careful analysis of the brine blowdown stream for sulfates is necessary to prevent calcium sulfate scaling of the membrane cells.

(5) The pH of the electrode stream must be maintained from 3.5 to 4.0 at the beginning of a cycle.

Table XI contains comparative data on three processes used experimentally by the Army to produce potable water for military use. Values given are estimates.

Table XI. Comparative Data

Process	Type of Water		Water Produced	
	Raw	Product	Gal. per lb Supplies	Lb per lb Supplies
	(mg/l TDS)			
Electrodialysis	3,500	500	32	270
	102	2	4	33
Ion Exchange	3,500	500	5.5	45
	102	2	160	1,330
Distillation	3,500	<10	18	150
	102	<10	18	150

It can be seen from the data in Table XI that the batch-type electrodialysis process produces more water per pound of supplies used than does either the ion exchange or distillation process when approximately 3,000 mg/l of ionized salts is removed from a brackish water containing 3,500 mg/l TDS. When 100 mg/l ionized salts is removed from a feed water containing 102 mg/l TDS, the ion exchange process is far superior to either the electrodialysis or the distillation process. The economical method for field demineralization of water would, therefore, seem to be a combination electrodialytic/ion exchange process operating in series when water of very low mineral content is desired as required in removal of radioactive contaminants.

IV. CONCLUSIONS

9. Conclusions. It is concluded:

a. The electrodialysis process (batch-type) is a more economical process than either ion exchange or vapor-compression distillation for removing dissolved minerals from a brackish water containing 3,500 mg/l TDS down to a drinkable level of 500 mg/l TDS.

b. Dissolved radioisotopes can be removed from water in high percentages by operation of the unit for complete demineralization, but it would not be practicable to do this from a military point of view, since the production rate would decrease sharply and concentration polarization may become significant.

c. Further laboratory and field tests using the electrodialysis process are necessary in order to develop simplified operating techniques, optimize performance, and adapt the process to military usage.

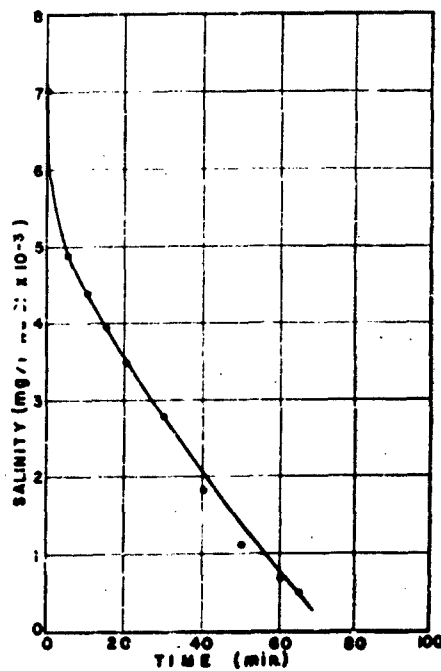
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GLOSSARY

1. Spacer - A plastic manifolded section used between membranes to control hydraulic flow of fluid (see Fig. 12).
2. Influent - That water entering the unit.
3. Effluent - That water leaving the unit.
4. Holes - Circular openings cut into the plastic spacers (see Fig. 11).
5. Melting - The changing of the chemical structure and physical appearance of a spacer (see Figs. 12 and 13).
6. μ curie/ml - micro-curie per milliliter or 10^{-6} curie/ml.
7. TDS - Total dissolved solids.
8. mg/l - milligrams per liter or parts per million, ppm.
9. mr/hr - milli-roentgens per hour or all the gamma radiation measured per unit of time.
10. mrem - milli-roentgen equivalent man or all ionizing radiation emitted (sum total).
11. $2 \text{ Cl}^- \rightarrow \text{Cl}_2 (\text{g}) + 2\text{e}^-$ This chemical equation states that two chloride ions are oxidized to chlorine gas with the gain of two electrons (equation b(1), paragraph 7).
12. Explanation of Graph Nomenclature - Example shown below.



The above graph is a plot of salinity on the ordinate versus time on the abscissa. At time - 40 minutes, the salinity (mg/l NaCl $\times 10^{-3}$) = 3.5. Hence, the salinity = 3.5×10^3 or 3,500 mg/l NaCl. This nomenclature is used throughout the report.

APPENDICES

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A	AUTHORITY	63
B	TEST DATA	67

APPENDIX A

AUTHORITY

SECURITY CLASSIFICATION UNCLASSIFIED

RESEARCH AND DEVELOPMENT PROJECT CARD (NEW PROJECTS) 2. SEC. U		3. PROJ. NO. 8-75-05-012
1. PROJECT TITLE WATER TREATMENT RESEARCH		4. CRG No. 2082/238
6. BASIC FIELD OR SUBJECT Water Supply		7. SUB FIELD OR SUBJECT SUB GROUP Filtration, Purification PO-7 Distillation
8. COMMANDANT - JENCV Corps of Engineers	12. CONTRACTOR AND/OR LABORATORY Engr. Res. & Dev. Laboratories Ft. Belvoir, Va.	CONTRACT/NO. NO.
9. DIRECTING AGENCY Engr. Res. & Dev. Div., HQ, OCE	13. RELATED PROJECTS 8-75-05-009 8-75-05-012 8-75-05-010 8-75-05-011	17. EST. EST. DATES RES. EST. DEV. TEST
10. REQUESTING AGENCY Office, Chief of Engineers	14. DATE APPROVED 7 November 1952 by GSUSA	OF. CVA. JUNE 1957
11. PARTICIPATION AND/OR COORDINATION Army Medical Service (C)	15. PRIORITY 2	16. FISCAL EST'S. 53 26M 54 10M 55 11M
20. REQUIREMENT AND/OR JUSTIFICATION: There is a requirement for certain research studies associated with the production of potable water by military engineering organizations. These studies are essential to establish the limitations of current water treating processes and equipment and the development of operational methods and techniques for producing drinking waters from natural sources which are considered difficult to treat because of their organic content.		
21. BRIEF OF PROJECT AND OBJECTIVE a. Brief: (1) Objective: (a) Investigate the hydraulic characteristics, and the chemical and mechanical limitations of solids contact clarifiers to provide design data and to improve performance and operational capabilities. (b) Investigate the limiting physical and chemical characteristics of pulverized limestone relative to its use as a coagulant aid in solids contact clarifiers. (c) Investigate the efficiency, effectiveness and handling problems associated with the use of water treatment chemicals needed for the purification of surface waters in the field to reduce cost, weight and bulk of required expendable supplies. (d) Investigate the disinfection requirements and methods used in conjunction with the water purification processes employing solids contact clarifiers and diatomite filters. * color content, temperature, chemical composition or sanitary quality. This project will provide valuable information and engineering data needed to improve the performance of new equipment recently developed and produce more efficient and economical operating methods.		
22. APPROVED BY	PC.	IC & P.
23. APPROVED BY	24. APPROVED BY	25. APPROVED BY

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PAGE 1 OF 1 PAGES

SECURITY CLASSIFICATION UNCLASSIFIED

SECURITY CLASSIFICATION UNCLASSIFIED

RESEARCH AND DEVELOPMENT PROJECT CARD (NEW PROJECTS)		1. PROJ. NO. 8-75-05-013
2. PROJECT TITLE WATER TREATMENT RESEARCH		3. Cognizant Agency CE 4. REPORT DATE 9 Oct. 52

(e) Investigate the effectiveness of chlorine as a disinfectant on highly colored waters.

(f) Investigate the influence of the mechanics of evaporation on the type and physical characteristics of scale formed in sea water evaporators.

(2) Military Characteristics:
Not appl cable.

b. Approach:

(1) A thorough investigation including laboratory and full scale comparative tests will be made to determine:

(a) Effects of slurry concentrations, flow rates, design and hydraulics of solids contact clarifiers using pulverized limestone for coagulation of natural surface waters.

(b) Effects of particle size, impurities and chemical composition of pulverized limestone when used as a coagulant aid in solids contact clarifier and its resultant effect on the chemistry of the effluent.

(c) Optimum methods of feeding, storage, handling, and packaging of water treating chemicals required for the purification of natural waters in the field.

(d) Limiting chlorine dosages and chlorine requirements when used as the disinfecting agent in water purification process employing clarification with Erdiator type solids contact clarifiers and filtration with diatomite filters.

(e) Efficiency of chlorine when used as the disinfecting agent in highly colored natural surface waters and the relationship between the color content and chlorine demand of the water.

(f) The influence of boiling rate and temperature, velocity of flow, heat exchange area, tube slenderness ratio and other currently unknown factors upon the type, hardness, K factor and brittleness of scales formed from normal sea water.

(2) Each of the investigations and studies listed above will be considered as a separate task under this project. As work is completed on each task, a report will be prepared covering the work accomplished, evaluating the findings and including such recommendations concerning equipment and methods as may be indicated.

c. Subtasks:

This project will be concerned with the problems which have become apparent during the development of equipment under the related projects, No. 8-75-05-009, Water Purification Equipment, Arctic, Set No. 1, Portable, 30 GPM; No. 8-75-05-010, Water Distillation Equipment, Non-Fouling; No. 8-75-05-011, Water Purification Equipment, Airborne, 600 GPM; and No. 8-75-05-012, Water Purification Unit, Electrified, Mobile, 1500 GPM.

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RESEARCH AND DEVELOPMENT PROJECT CARD (NEW PROJECTS)		SEC. U	PROJECT NO. 8-75-54-013
1. PROJECT TITLE		Sponsoring Agency	
WATER TREATMENT RESEARCH		Contract No. 52	
<p>The subjects to be studied are areas in which available information is incomplete, inadequate, or non-existent. This project, therefore, must provide design data and other information which has been demonstrated to be required if current equipment and developments are to be later improved.</p>			
d. Other Information:			
(1) References:			
None			
(2) Discussion:			
(a) Agencies interested in this project, in addition to the Corps of Engineers, with which liaison will be maintained, and which will be furnished copies of the reports on the project are:			
Department of the Air Force, Department of the Navy, the Army Medical Service, the Chemical Corps,			

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 SECURITY CLASSIFICATION UNCLASSIFIED

FORM 10-1-1963

APPENDIX B

TEST DATA

Table XII. Feed Salinity - 7080 mg/l NaCl,
9 Aug 1958 (Fig. 11)

Time (min)	Performance Data	
	AT (amps)	AB (amps)
3	3.02	2.00
5	-	1.87
7	3.01	1.85
9	-	1.75
12	2.98	1.60
15	2.95	1.78
17	-	1.75
20	2.89	1.41
22	-	1.45
25	2.82	1.40
28	-	1.52
30	2.77	1.44
35	2.69	1.34
40	2.60	1.25
50	2.40	0.85
55.5	2.23	0.82
60	2.12	0.81
65	1.95	0.77
70.5	1.78	0.71
75	1.60	0.75
80	1.41	0.64
85	1.21	0.64
88.8	1.04	0.75
Discharge	-	-

Table XIII. Rate of Demineralization
(Fig. 14)

Time (min)	Salinity (mg/l Na Cl)
0	7040
5	4878
10	4390
15	3950
20	3510
30	2810
40	1860
50	1125
60	705
64.4	538
Discharge	-

Table XIV. Effect of Salinity on Power
Consumption (Fig. 15)

Feed Salinity (mg/l Na Cl)	Power Consumption (kw-hr)	Time (hr)	Power Consumption (kw-hr/24-hr day)
1,650	2.5 ± 0.2	$6.5 \pm .05$	8.5 ± 0.7
1,770	2.4 ± 0.2	$7.2 \pm .05$	8.0 ± 0.7
2,515	6.7 ± 0.4	$17.5 \pm .05$	9.2 ± 0.7
3,580	4.4 ± 0.2	$12.3 \pm .05$	8.6 ± 0.4

Table XV. Electrode Stream pH Behavior
during a Typical Cycle (Fig. 16)

Time (min)	Electrode Stream pH at 68° F
2	6.6
9	5.4
12	3.7
15	3.3
18	3.2
20	3.2
25	3.2

Table XVI. Effect of Feed Water Salinity on Production Rate (Fig. 17)

Feed Salinity (mg/l Na Cl)	Product Flow Rate at 70° F (gph)
1,650	84.5
1,770	69.5
2,515	45.9
3,580	31.8
5,067	25.7
7,078	19.7
9,004	15.1
12,070	11.0

Table XVII. Effects of Iron Sliming and Acid Cleaning on Production Rate (Fig. 26)

Date	Temp. Avg. (° F)	Production Rate (gph)		Operating Hours	
		T	70° F	Differential	Cumulative
30 Sept	75	63.4	59.9	7.1	7.1
1 Oct	74	55.0	52.6	14.3	21.4
2 Oct	74	51.7	49.4	3.1	24.5
3 Oct	72	47.7	46.6	14.8	39.3
5 Oct	70	42.2	42.2	5.8	45.1
6 Oct	75	44.7	42.2	6.9	52.0
7 Oct	72	40.2	39.3	7.9	59.9
8 Oct	75	37.3	35.2	7.5	67.4
16 Oct	80	69.7	62.0	4.7	72.1
17 Oct	70	55.7	55.7	2.3	74.4
19 Oct	72	51.4	50.3	4.3	78.7
20 Oct	76	65.1	60.8	7.5	86.2
21 Oct	68	66.4	67.9	4.5	90.7
	78	76.2	69.5	2.5	93.2
22 Oct	76	72.9	68.1	7.2	100.4

Table XVIII. Cycle No. 4,
29 October 1959 (Fig. 27)

Time (min)	Direct Current (m amps)
0	580
3	540
7	493
15	400
25	290
36	195
45	94
75	50
85	41
98.7*	32

* Discharge of product.

Table XIX. Rate of Demineralization
(Fig. 28)

Passes through Stack	TDS, mg/l
0	900
1	540
2	324
3	194
4	116
5	70
6	42
7	25
8	15
9	9
10	5.4
11	3.2
12	1.9
13	1.1
14	0.7

Table XX. Effect of TDS Removal on Power Consumption
(Fig. 29)

Time (hrs)	TDS in Product (mg/l)	Temp. Avg. (° F)	Power Consumption (kw-hr/1000 gal)	
			T	60° F
-	880	-	0	0
100.4	311	74	12	14
28.1	5*	57	52	50
11.2	1.5*	51	90	81

* As Na Cl, using Barstead Purity Meter.

Table XXI. Cs¹³⁷ Concentration Decrease in Feed Tank
during Decontamination Study (Fig. 30)

Date	Average Cs ¹³⁷ Concentration (uc/ml)
28 Oct	1238 x 10 ⁻⁵
29 Oct	-
30 Oct	965
31 Oct	883
2 Nov	781
3 Nov	710

Table XXII. Typical Decontamination Cycle
(Fig. 31)

Time (min)	Cs ¹³⁷ Concentration $\mu\text{c/ml} \times 10^5$	
	Product Stream	Brine Stream
0	1,238	-
5	411	1,970
10	319	1,920
20	174	1,840
40	54	1,600
60	16	1,500
80	7	1,320
98.7(a)	1	1,310
108.4(b)	-	1,238

(a) End of cycle.

(b) Discharge of product.

Table XXIII. Chemical Analysis of Typical Demineralization Run
(Figs. 27, 35, 36, 37, 38)

Date and Sample No.	Ca	Mg	Fe	Cl	HCO ₃	SO ₄	SiO ₂	TDS	pH	Calcium CaCO ₃	Magnesium CaCO ₃
Brine, #4, 29 Oct 59											
B	204.	27.5		31.9	0	1,062		1,460		511	113
C	188.5	23.1		29.8	2.6	1,000		1,377		471	95
D	154.4	19.5		31.9	15.2	925		1,293		386	80
E	105.2	14.6		27.0	8.8	788		1,079		263	60
F	88.0	12.2		24.8	3.7	692		942		220	50
G	80.0	10.7		22.7	1.3	600		818		200	44
H	76.8	9.7		19.9	0	540		735		192	40
Product, #4, 29 Oct 59											
A	84.0	10.2	0.8	18.5	53.6	522	(64)	788		-	42
B	46.4	8.0		9.9	33.2	248		390		-	-
C	35.5	6.7		8.5	26.0	216		320		88.8	27.2
D	22.4	4.3		4.2	20.4	135		212		-	-
E	7.7	1.8		0.7	13.6	+3		77		19.2	7.6
F	2.6	0.8		0.7	8.7	15		33		-	-
G	0.6	0.3		0.7	4.8	-		10.0		1.6	1.2
H	-	-		-	1	-		5.0		-	-

Table XXIV. Operation of Electrodialysis Unit for Total Demineralization

Date	Time (hr)	Cord. Setting (μ mhos/cm)	Prod. Rate (gph)	Rate (gal)	Brine Rate (gph)	Elect. Rate (gph)	Total Water (gal)	TDS (mg/l as NaCl)	Temp. (avg.) (° F)
28 Oct	7.0	20	14.6	102	21.5	151	403	5.0	66
29 Oct	7.3	20	12.8	93	23.4	171	425	5.0	59
30 Oct	6.8	20	9.9	67	18.0	122	349	5.0	50
31 Oct	7.0	20	9.7	66	19.8	139	371	4.8	51
2 Nov	7.4	0	6.1	45	20.2	149	360	1.5	52
3 Nov	3.8	0	5.9	22	19.6	75	184	1.5	51

Table XXV. Demineralization Analyses: NTS, 1959 Electrodialysis Unit

Time (hr)	Date	Ca	Mg	Fe	Cl	HCO ₃	SiO ₂	SO ₄	TDS	pH
7.1	30 Sept	23	4.0	0.75	8	25	64	165	35	6.9
14.3	30 Sept-1	23	3.6	0.75	9	30	64	153	344	7.2
3.1	2 Oct	25	2.9	0.6	6	31	64	113	283	7.4
14.8	2-3 Oct	15	2.2	0.6	7	29	64	122	293	-
5.8	5 Oct	19	2.8	0.5	8	29	64	110	285	7.2
6.9	6 Oct	-	-	-	-	-	-	-	-	-
7.9	7 Oct	11	2.8	0.5	-	16	64	110	261	7.2
7.5	8 Oct	15	2.8	0.6	5	26	64	155	332	7.3
4.7	16 Oct	21	3.3	0.65	9	25	64	138	314	6.8
2.3	17 Oct	17	2.9	0.65	9	31	64	118	300	7.4
4.3	19 Oct	-	-	-	-	-	-	-	-	-
7.5	20 Oct	20	3.4	0.5	8	26	64	122	298	7.3
7.0	21 Oct	24	3.6	0.5	6	25	64	153	333	7.2
7.2	22 Oct	23	3.7	0.5	6	25	64	150	329	7.2
	Average	19	3.2	0.6	7	26	64	134	311	7.2
	Raw Water	82.4	12.4	1.3	18.1	53.7	64	528	889	7.8

Category 16 - Water Supply and Sanitation

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Electrodialysis

DATE OF REPORT 9 May 1961 PROJECT SM75-05-001-06 CLASSIFICATION Uncl.

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<p>10 U. S. Army Research Development and Development Laboratories, Fort Belvoir, Virginia - WATER DEMINERALIZATION AND DECONTAMINATION STUDIES UTILIZING ELECTRODIALYSIS - Paul E. Decker, Jr.</p> <p>Report 107-28, 9 May 61, 73 pp, 30 illus, 25 tables NA Project 087-05-001-06 Unclassified Report</p> <p>Report covers laboratory and field tests of an electrolysis process for removal of limited salts from brackish waters. The unit tested was a Model 3-4 procured from J. W. Carr, Inc., Cambridge, Mass. Laboratory tests were conducted at Fort Belvoir to determine the following: (1) suitability of operational features to military usage; (2) performance characteristics of process; (3) effect of changes in temperature, pH, and feed salinity on performance; and (4) factors reducing effectiveness of the ion-selective membranes. Tests were conducted in the field to determine the behavior of the demineralizer in the field when removing dissolved mineral matter from brackish well water and to determine the capability of the unit for removal of soluble radioisotopes from water. The report concludes: (a) the electrolysis process (batch-type) is more economical than either ion exchange or vapor-compression distillation for removing dissolved minerals from a brackish water containing 3,500 mg/l TDS down to a drinkable level of 500 mg/l TDS; (b) dissolved radioisotopes can be removed from water in high percentages by operation of the unit for complete demineralization, but it would not be practicable to do this from a military point of view, since the production rate would decrease sharply and concentration polarization may become significant; and (c) further laboratory and field tests using the electrolysis process are necessary in order to develop simplified operating techniques, optimize performance, and adapt the process to military usage.</p>	<p>UNCLASSIFIED 1. Water Supply - Filtration, Purification, Distillation 2. Contracts - None</p>	<p>10 U. S. Army Research Development and Development Laboratories, Fort Belvoir, Virginia - WATER DEMINERALIZATION AND DECONTAMINATION STUDIES UTILIZING ELECTRODIALYSIS - Paul E. Decker, Jr.</p> <p>Report 107-28, 9 May 61, 73 pp, 30 illus, 25 tables NA Project 087-05-001-06 Unclassified Report</p> <p>Report covers laboratory and field tests of an electrolysis process for removal of limited salts from brackish waters. The unit tested was a Model 3-4 procured from J. W. Carr, Inc., Cambridge, Mass. Laboratory tests were conducted at Fort Belvoir to determine the following: (1) suitability of operational features to military usage; (2) performance characteristics of process; (3) effect of changes in temperature, pH, and feed salinity on performance; and (4) factors reducing effectiveness of the ion-selective membranes. Tests were conducted in the field to determine the behavior of the demineralizer in the field when removing dissolved mineral matter from brackish well water and to determine the capability of the unit for removal of soluble radioisotopes from water. The report concludes: (a) the electrolysis process (batch-type) is more economical than either ion exchange or vapor-compression distillation for removing dissolved minerals from a brackish water containing 3,500 mg/l TDS down to a drinkable level of 500 mg/l TDS; (b) dissolved radioisotopes can be removed from water in high percentages by operation of the unit for complete demineralization, but it would not be practicable to do this from a military point of view, since the production rate would decrease sharply and concentration polarization may become significant; and (c) further laboratory and field tests using the electrolysis process are necessary in order to develop simplified operating techniques, optimize performance, and adapt the process to military usage.</p>
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